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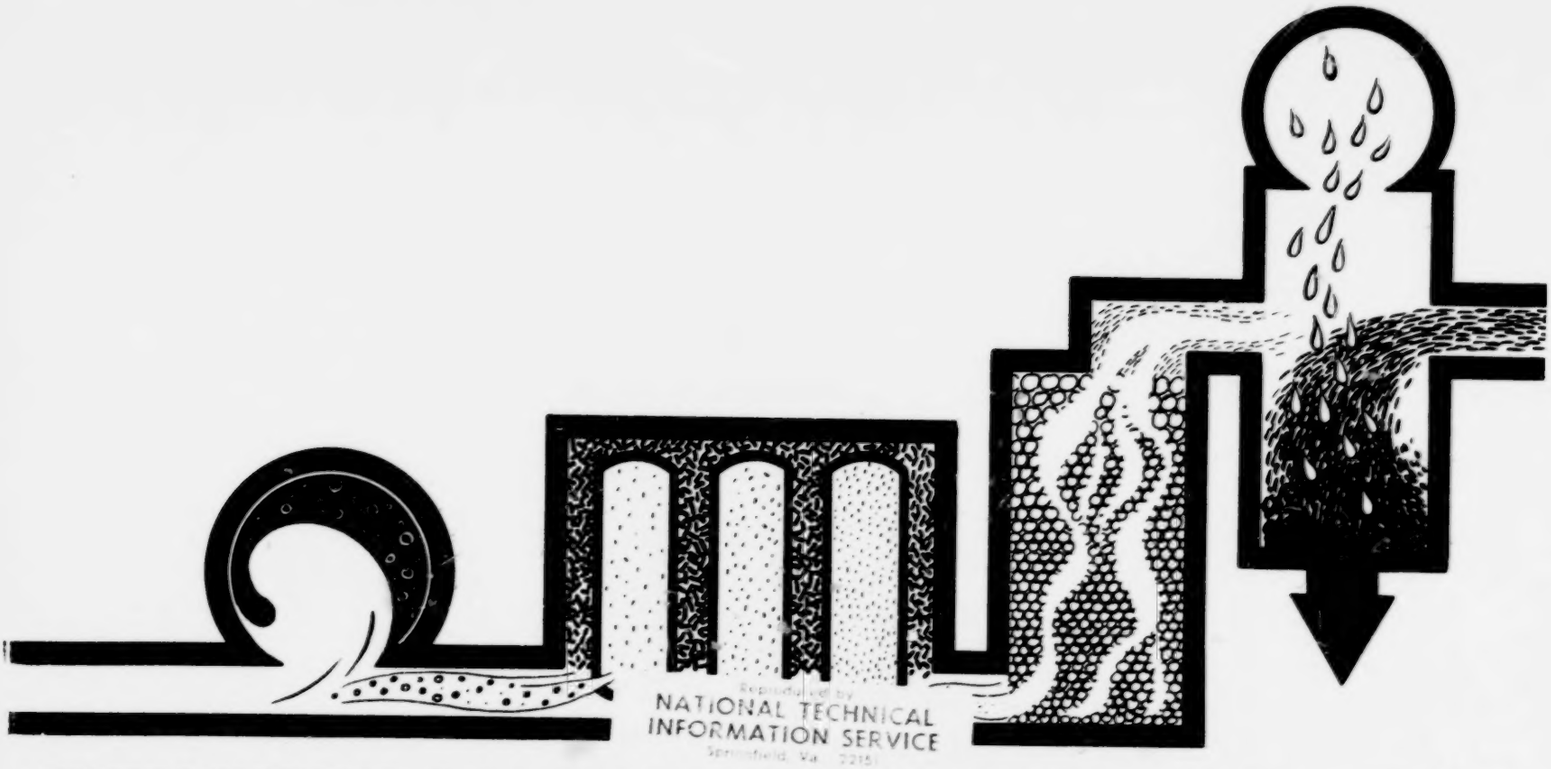
RENOVATION OF MUNICIPAL WASTEWATER BY REVERSE OSMOSIS



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27 Abstract The three major configurations of reverse osmosis units include spiral wound units, tubular units, and plate and frame units. Tests were conducted on prototype units employing all three configurations, and the membranes were found capable of rejecting 93 to 95% of TDS, 90 to 99% of phosphates, 80 to 90 % of ammonia nitrogen, 60 to 70% of nitrate nitrogen, 99 to 100% of particulate matter, 90 to 95% of TOC and greater than 90% of COD. Many of the problems discovered stemmed from the use of prototype units which had not been thoroughly tested and were subject to repeated mechanical failures. Fouling problems have been partially alleviated by periodically depressurizing the membranes and washing them with enzyme detergents. The success of this method is attributed to protein hydrolysis of the slime layer which coats the membrane. Increasing population has dictated the recycling of a much larger portion of the nation's water. Each time it is re-used, the water picks up greater amounts of dissolved solids. These solids will eventually have to be removed to preserve the potability of drinking water. At present, reverse osmosis techniques are the most promising methods for dissolved solids removals. Also, increasing advances in membrane materials have steadily decreased the cost of reverse osmosis to the point that a reverse osmosis system coupled with primary and sand filtration only has an estimated cost of 35.4¢/1000 gallons. This figure is equal to the cost of conventional activated sludge treatment plus filtration and carbon adsorption. (Lowry-Texas)

Abstractor Mark V. Lowry | Institution Texas Univ, Austin

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RENOVATION OF MUNICIPAL WASTEWATER BY REVERSE OSMOSIS

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ABSTRACT

The increasing demand for clean water makes clear the need for improved treatment of municipal wastewater. Deliberate reuse of wastewater requires eificient removal of "residual" organic and inorganic solutes. A common and effective approach has been to supplement biological processes with chemical and physical processes that, together, are capable of achieving water quality consistent with reuse requirements. A more direct approach is to use a single process such as reverse osmosis which exhibits a potential to separate a broad spectrum of contaminants from primary and secondary effluents.

The pilot plant studies reported herein were addressed toward defining the problems associated with the reverse osmosis processing of variously treated wastewaters. The effects of feed water quality on membrane performance were evaluated, as were methods to control membrane fouling. A comparison of three configurations: flat plate, spiral-wound, and tubular was made to determine their relative merit in treating wastewater. Finally, a cursory examination of costs was made.

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INTRODUCTION

Since the fresh water resources of the world are limited, it is axiomatic that our ever-increasing water needs will demand more efficient use of our present water supplies.

One way of improving this efficiency is through multiple use of available surface water. This, of course, requires treatment technology capable of achieving water quality consistent with the planned future uses of the water. In many cases, present-day treatment techniques will not be adequate, and must be supplemented or replaced with advanced processes that can effect a high removal of undesirable contaminants. Of primary concern is the removal of nutrients, dissolved mineral salts, oxygen-demanding organics and undesirable bacteria and viruses presently being discharged as a result of inadequate wastewater treatment.

Advanced waste treatment of primary or secondary municipal effluents is currently accomplished by a series of unit processes, each capable of selectively removing a particular fraction of the residual waste load. This approach has been successful but could be enhanced through the development and utilization of a single process exhibiting removal efficiencies of the combined unit processes.

Reverse osmosis is a process with the demonstrated capacity to separate dissolved organics and inorganics as well as the larger organic species from aqueous solution, and is therefore worthy of evaluation as a potential advanced waste treatment process.

HISTORY OF REVERSE OSMOSIS DEVELOPMENT

The early work of Reid and co-workers at the University of Florida first demonstrated the effectiveness of cellulose acetate as a desalination membrane (1). Further investigation by Loeb and Sourirajan (1959-1960) at the University of California, using aqueous magnesium perchlorate as a swelling agent in the casting solution for cellulose acetate membranes, resulted in a membrane that exhibited much higher water permeation rates while still maintaining the solute rejection characteristics of the earlier membranes. The original Loeb-Sourirajan film formulation, identified as batch No. 25, is shown in Table 1 (2)(3)(4).

TABLE 1

CASTING SOLUTION BATCH NO. 25

COMPONENT	(WT %)
Cellulose Acetate (acetyl content 39.8%)	22.2
Acetone	66.7
Magnesium Perchlorate	1.1
Water	10.0
TOTAL	100.0

This formulation established the practicality of reverse osmosis as a desalination process, and has been reported extensively in the literature. Innumerable modifications of the early Loeb formulation have been made since 1960 in an effort to improve the initial flux and solute rejection characteristics of the membranes. These modifications have included basic changes in the casting formulation as well as improvements in the casting and curing techniques. The basic cellulose acetate designated by Eastman Kodak as E-398-3 is now used in combination with additives and other cellulosic derivatives to produce optimum membranes for the conversion of brackish and sea water. Much of the membrane development work to date has been sponsored by the U. S. Department of the Interior's Office of Saline Water.

As the development of membranes progressed, the need for a practical module configuration became apparent and the need was met by the design and testing of three principal configurations; the plate and frame, the spiral-wound, and the tubular modules. Extensive pilot plant evaluation of these modules on brackish water illuminated the problems of concentration polarization and scaling that could be expected with these feeds. The effect of concentration polarization was minimized by maintaining turbulent flow conditions within the brine channels while the precipitation of sparingly soluble salts was controlled by depressing the feed stream pH to a value of 6.0 or less.

The application of reverse osmosis in treating municipal wastes was first considered late in 1962 when preliminary investigation by Aerojet-General Corporation at Azusa, California indicated that the cellulose acetate membranes were an effective barrier for both organic and inorganic solutes. Following preliminary testing, a contract was awarded to Aerojet by the Public Health Service's Advanced Waste Treatment Program (now FWQA) to conduct an in-depth bench-scale study to evaluate the potential of reverse osmosis for renovation of municipal wastes.

This study was conducted on filtered municipal secondary effluent using 3-inch diameter flat-plate test cells operated at pressures of 750 and 1500 PSIG. The feed stream was recirculated during the tests and a 5-10 fold increase in feed concentration was produced. The pH of the feed stream was maintained at 5.5 by the addition of sulfuric acid. Three types of cellulose acetate membranes were used for these tests with nominal flux rates that varied from 10 to 60 gallons per square foot per day (GFD). Results of this study demonstrated that high quality water could be produced by this process from secondary effluent. Results of two tests are shown in Table 2 and indicate greater than 95% removal of total dissolved solids (TDS) and chemical oxygen demand (COD)(5).

It was also found that high flux (30-60 GFD) membranes were unstable and, after a few hours of operation, exhibited flux rates as low as the lower flux (10-20 GFD) membranes. Generally, the flux rates were considered too low to be practical. Several possible causes of the high flux rates were proposed, including the possibility of organic fouling, poor hydrodynamic conditions and excessive concentration of organic solutes. There was also some question regarding the resistance of the membranes to biological attack.

This initial bench-scale evaluation using the flat-plate test cells was followed in early 1965 by a series of experiments conducted at the Pomona Pilot Plant located adjacent to the Pomona Water Renovation Plant in Pomona, California. This study was a cooperative effort between the County Sanitation Districts of Los Angeles County and the

TABLE 2

WATER MINERAL ANALYSIS
INITIAL BENCH SCALE STUDY
AEROJET-GENERAL CORPORATION

	Feed Water (Typical) ppm	Product (Test 18) ppm	Product (Test 28) ppm
TDS	550	15	28
ABS	4.5	0.1	0.1
COD	95	2	6.0
pH	5[1]	6.3	5.5
Cations			
Na^{+}	85	4.6	6.1
K^{++}	40	4.9	2.7
NH_4^{+}	25	0	3.9
Ca^{++}	125	0	2.6
Mg^{++}	50	0.7	0
Fe^{++}	N.D.	0	0
Anions			
Cl^{-}	65	20.3	22.1
NO_3^{-}	2	0	0
HCO_3^{-}	260	8.1	3.5
$CO_3^{=}$	0	0	0
$SO_4^{=}$	200	1.7	3.3
$SiO_3^{=}$	30	2.9	2.9
$PO_4^{=}$ (total)	25	0	0

N.D. - not determined

1 - pH adjusted during test.

General Atomic Division of the General Dynamics Corporation (5). The experiments were conducted at a pressure of 200 PSIG at 5% recovery using the spiral-wound modules developed by General Atomic. The feed to the test loop was diatomaceous earth-filtered secondary effluent.

Results of these tests again demonstrated the high organic and inorganic solute rejection seen in the earlier tests. Also noted during these tests was a significant decrease in membrane flux with time. This was attributed to fouling of the membranes by deposition of organic material present in filtered secondary effluent. This deposit could be partially removed by increasing local turbulence at the membrane surface and by establishing a backward flow potential through the membrane.

These preliminary results, while promising, clearly indicated the need for continuing the investigation of reverse osmosis at the pilot plant scale to further evaluate the fouling potential of various treated waste streams, to determine optimum operating conditions, to examine different module configurations, and to determine the required pretreatment for sustained operation.

PILOT PLANT INVESTIGATIONS

(Pomona, California)

Spiral-Wound Configuration

The first pilot plant investigation of reverse osmosis was conducted at the Pomona Water Renovation Plant under a joint FWQA-LACSD contract in 1966. This study was conducted using a 5000 GPD unit employing the spiral-wound module shown in Figure 1. The module consists of 3/8" PVC pipe to which is attached a permeate flow channel enclosed on each side by a cellulose acetate membrane. The membrane is glued around the edge to form an envelope through which the permeate flows to the center collection pipe. The feed enters the module axially and is distributed through the module by a polyethylene vexar brine-side spacer. This spacer is rolled up between the membranes to form a cylindrical module which is then placed into the pressure vessel. A completed module is shown in Figure 2. The arrangement of the individual modules within a pressure vessel is shown in Figure 3, and Figure 4 is a flow diagram of the entire unit.

The first 11 months of operation of the spiral-wound module on secondary effluent were plagued by problems of module construction and mechanical failures. The major problems were excessive module leaking, brine seal failures, and telescoping of modules. These problems were compounded by a faulty pH control system and frequent pump failures.

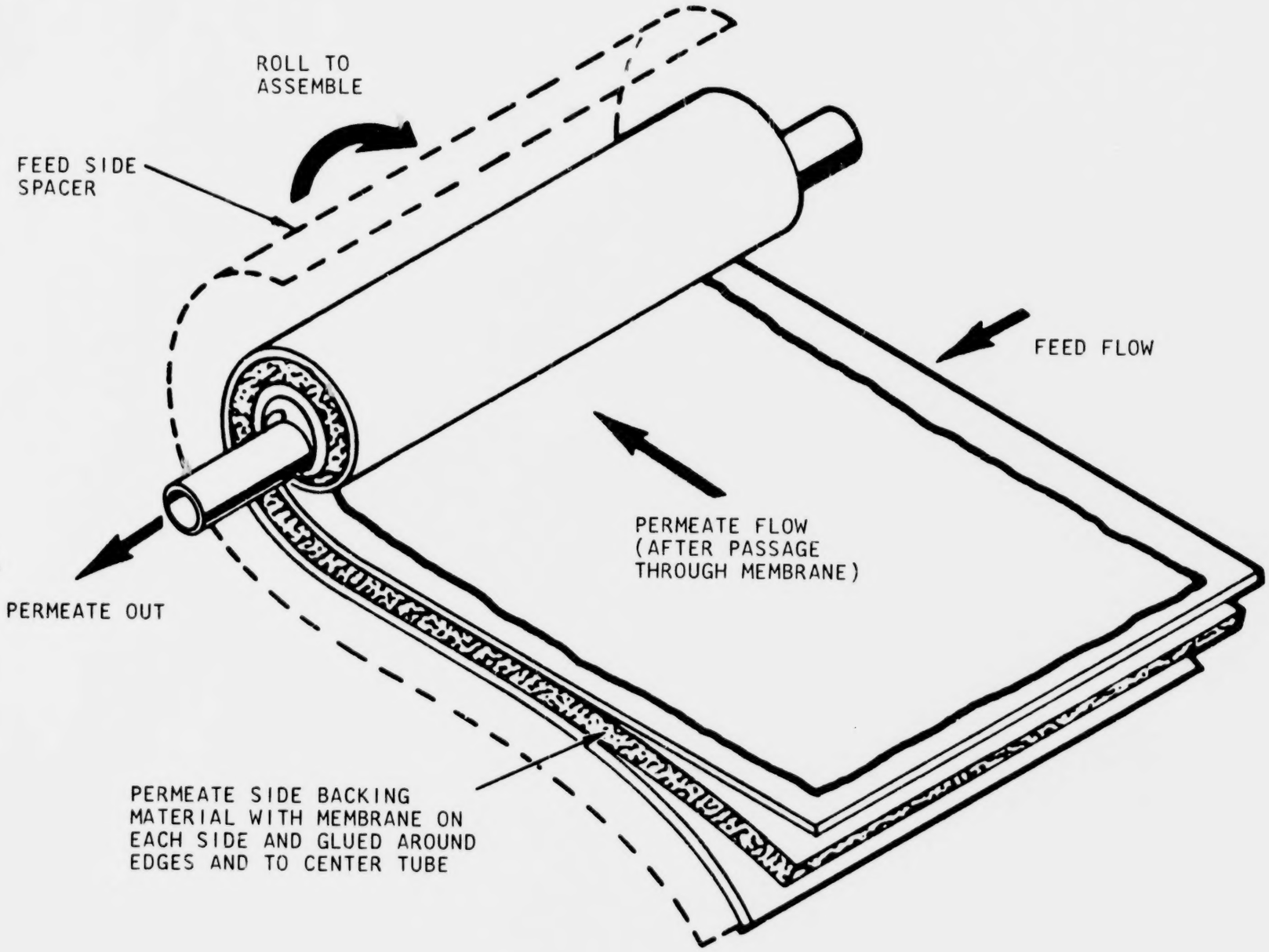
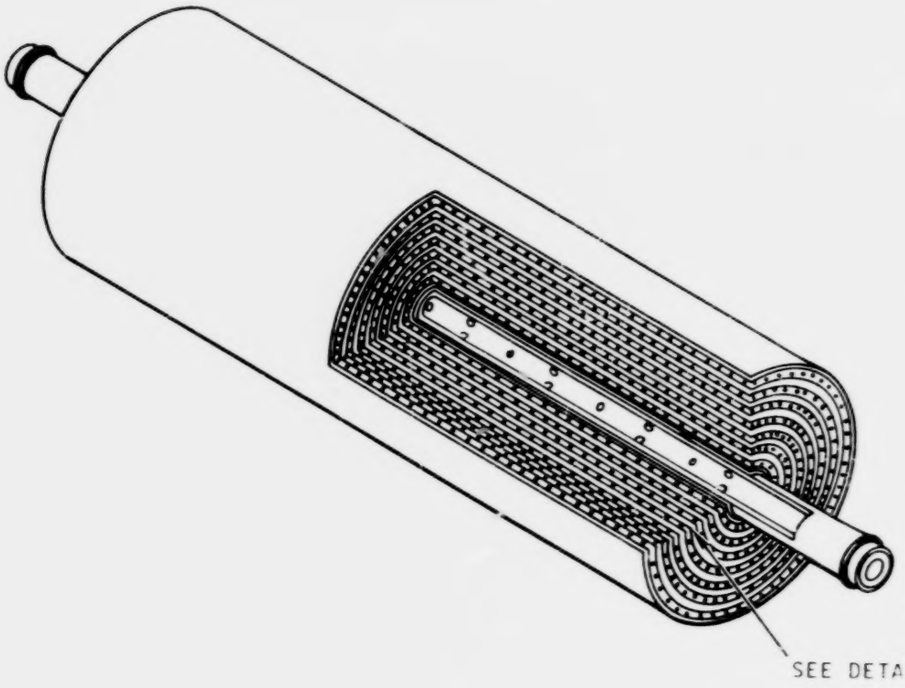


FIGURE 1. REVERSE OSMOSIS MODULE ASSEMBLY



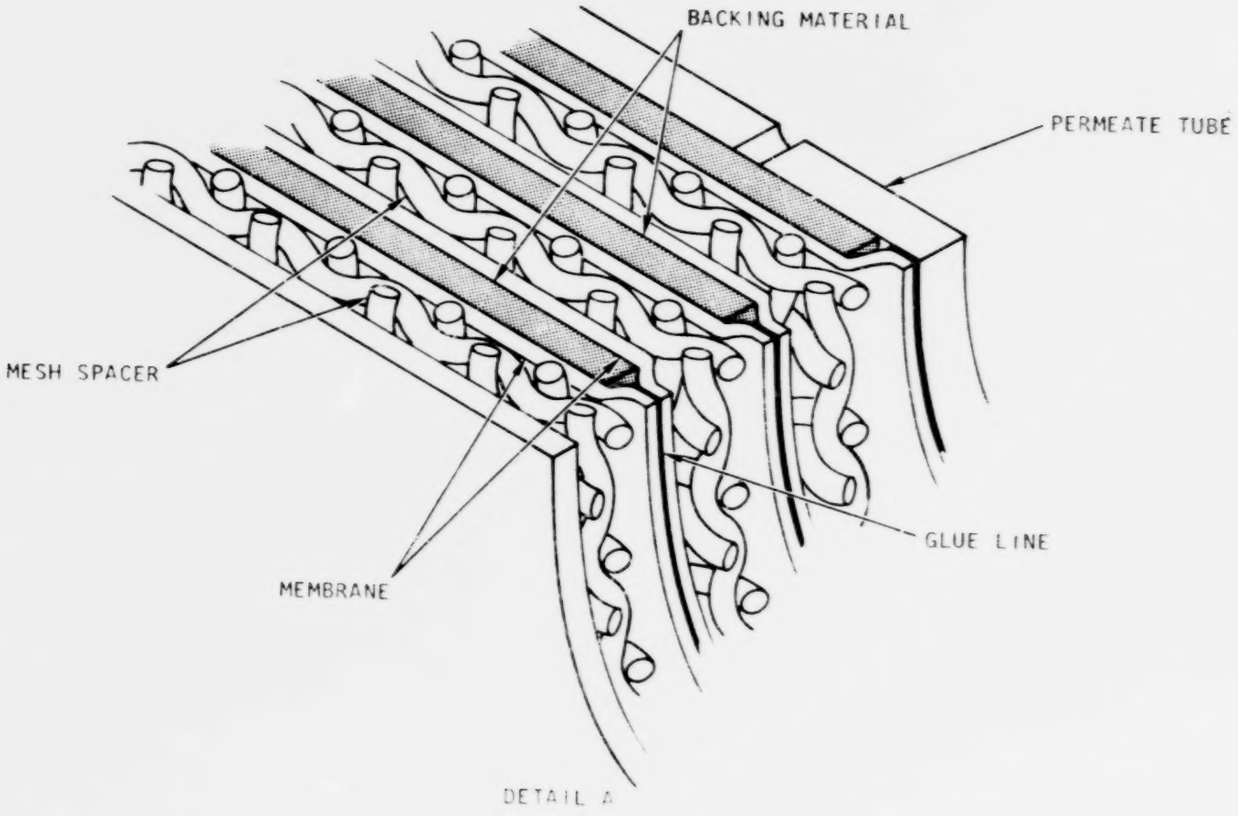


FIGURE 2. MODULE FABRICATION

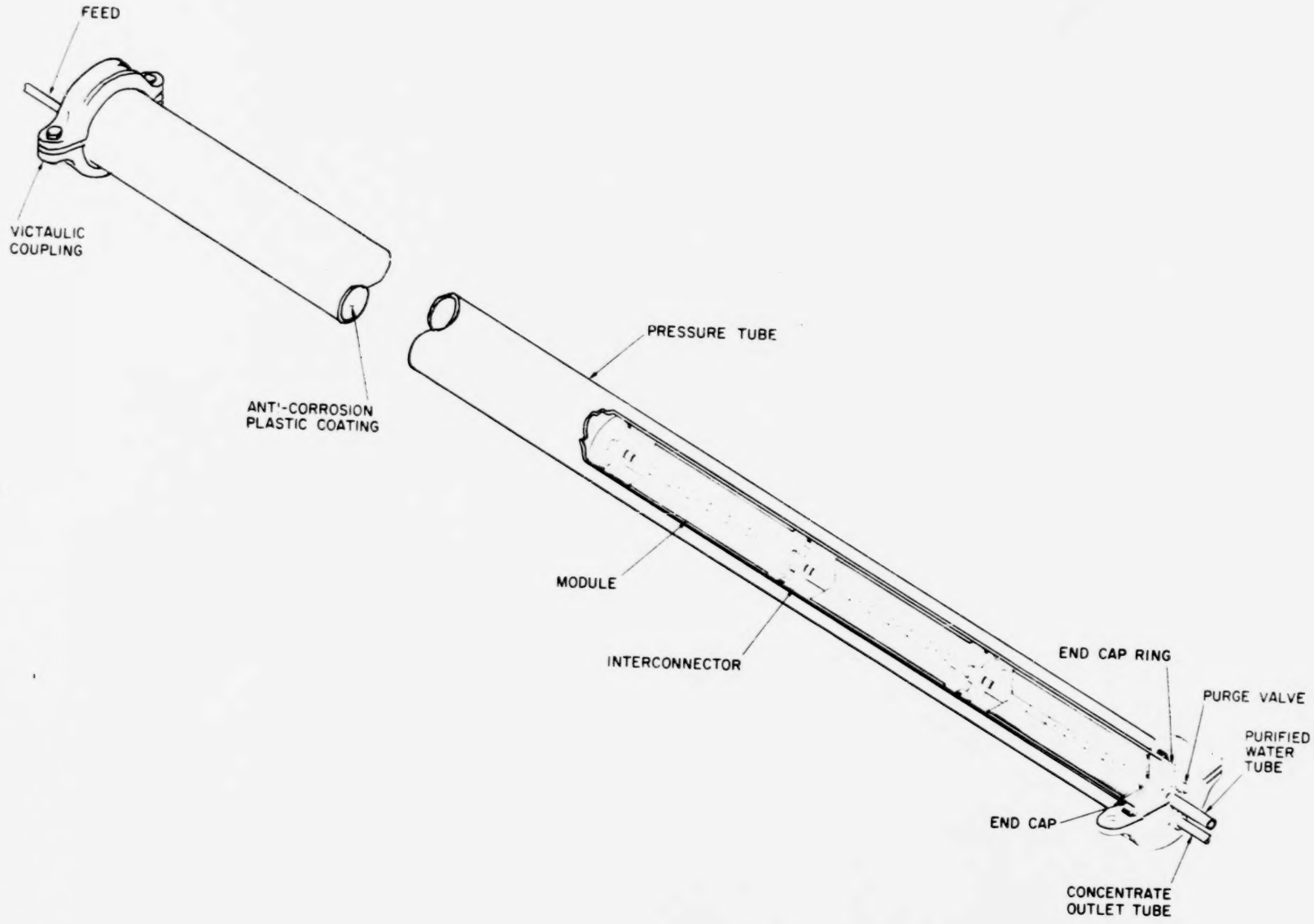


FIGURE 3. ARRANGEMENT OF SPIRAL-WOUND MODULES IN A MULTIMODULE PRESSURE VESSEL

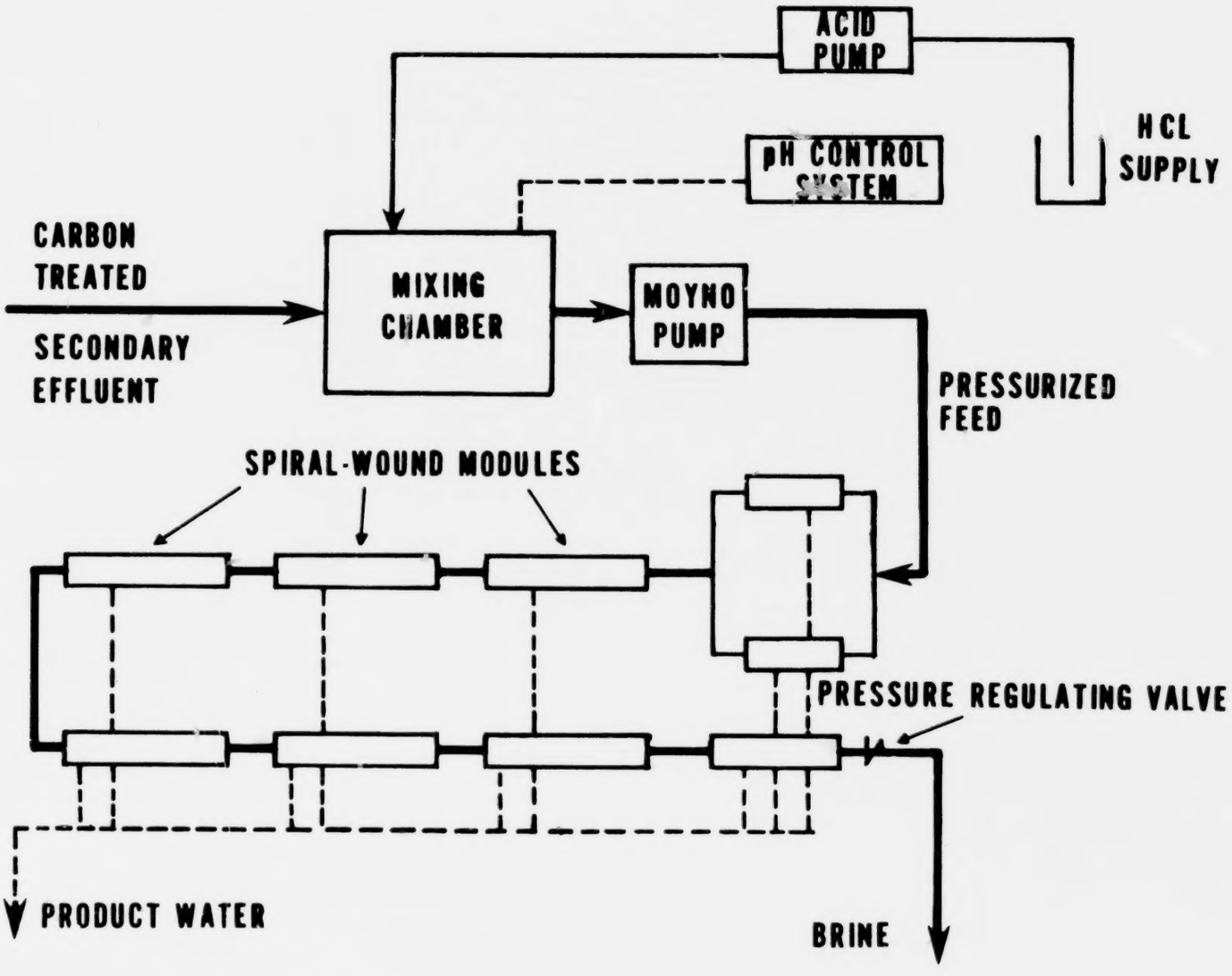


FIGURE 4

FLOW DIAGRAM OF GULF GENERAL ATOMIC

REVERSE OSMOSIS TEST FACILITY, POMONA, CALIFORNIA

Notwithstanding these early difficulties, it was concluded that the spiral-wound modules could process secondary effluent with high organic and inorganic rejection, but that some form of pretreatment was necessary to prevent excessive clogging of the brine-side spacers from suspended solids present in secondary effluent.

Bearing this in mind, and considering the possible fouling potential of soluble organic species, it was decided to pretreat the reverse osmosis feed by passage through a granular carbon adsorption process.

Two runs were conducted on the carbon-treated secondary effluent. Objectives of these two runs are shown below:

1. To determine the water-flux-time relationship for the system when operating on an effluent from an activated carbon plant.

2. To monitor the water quality produced by the membranes and note any change in water quality with time.

3. To achieve a high rate of water recovery from the system and to determine, if possible, any adverse effects on the system as a result of high-water recovery.

4. To test the structural integrity of spiral-wound modules being subjected to a long-term test under normal environmental conditions.

5. To determine the materials responsible for any membrane fouling which might occur.

6. To investigate procedures for minimizing or preventing membrane fouling, along with methods for cleaning fouled membranes.

A summary of the operating conditions for the two experimental runs are shown in Table 3. It should be noted that for Run #1 the diameter of modules within the seven series pipes are progressively decreased to provide a relatively constant brine velocity, while Run #2 was conducted at a higher initial, but gradually decreasing, brine velocity since all of the modules are 4-inches in diameter.

TABLE 3

SUMMARY OF CONDITIONS FOR EXPERIMENTAL
RUN IN GULF GENERAL ATOMIC REVERSE OSMOSIS UNITS

	Run #1	Run #2
No. of Modules in Unit	85	68
Module Diameter	45-4", 20-3", 20-2"	All 4"
Total Membrane Area	550 ft^2	632 ft^2
Feed Water Source	Carbon Effluent	Carbon Effluent
Feed Pressure	400 psi	400 psi
Initial Feed Flow	2.8 gpm (4040 gpd)	4.8 gpm (6910 gpd)
Initial Brine Flow	0.56 gpm (805 gpd)	1.0 gpm (1440 gpd)
Initial Water Recovery	80%	80%
pH Control	to 5.0 using HCl	to 5.0 using HCl
Chlorination	5.0 mg/l dosage using NaOCl	5.0 mg/l dosage using NaOCl

A summary of the data from each experiment is shown in Figures 5 and 6. These figures show the time dependent variation of flux and salt rejection throughout the run. Salt rejection was calculated for these runs by use of the formula:

$$\text{Salt rejection} = \frac{\text{Average feed conductivity} - \text{Product conductivity}}{\text{Average feed conductivity}}$$

where,

$$\text{Average feed conductivity} = \frac{\text{Feed conductivity} + \text{brine conductivity}}{2}$$

All data was obtained from readings taken daily and then averaged over 100-hour intervals.

The flux decline curve for Run #1 can be divided into 3 segmerts - 0 - 600 hours, 700 - 1600 hours, 1700 - 2500 hours. The abrupt increase in flux rate between 600 and 700 hours is due principally

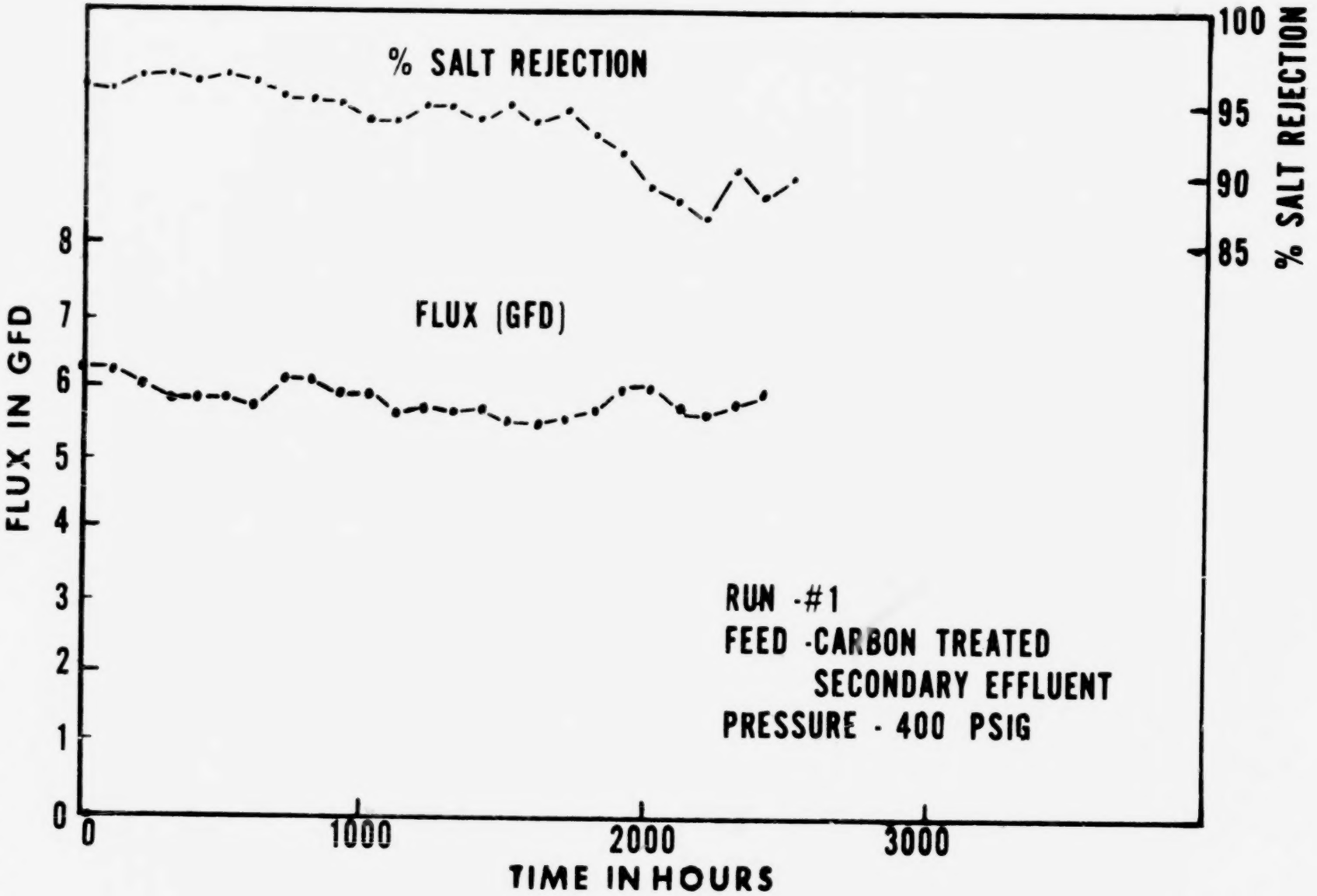


FIGURE 5
FLUX AND SALT REJECTION PERFORMANCE FOR GULF GENERAL
ATOMIC SPIRAL-WOUND REVERSE OSMOSIS MODULE, POMONA, CALIFORNIA

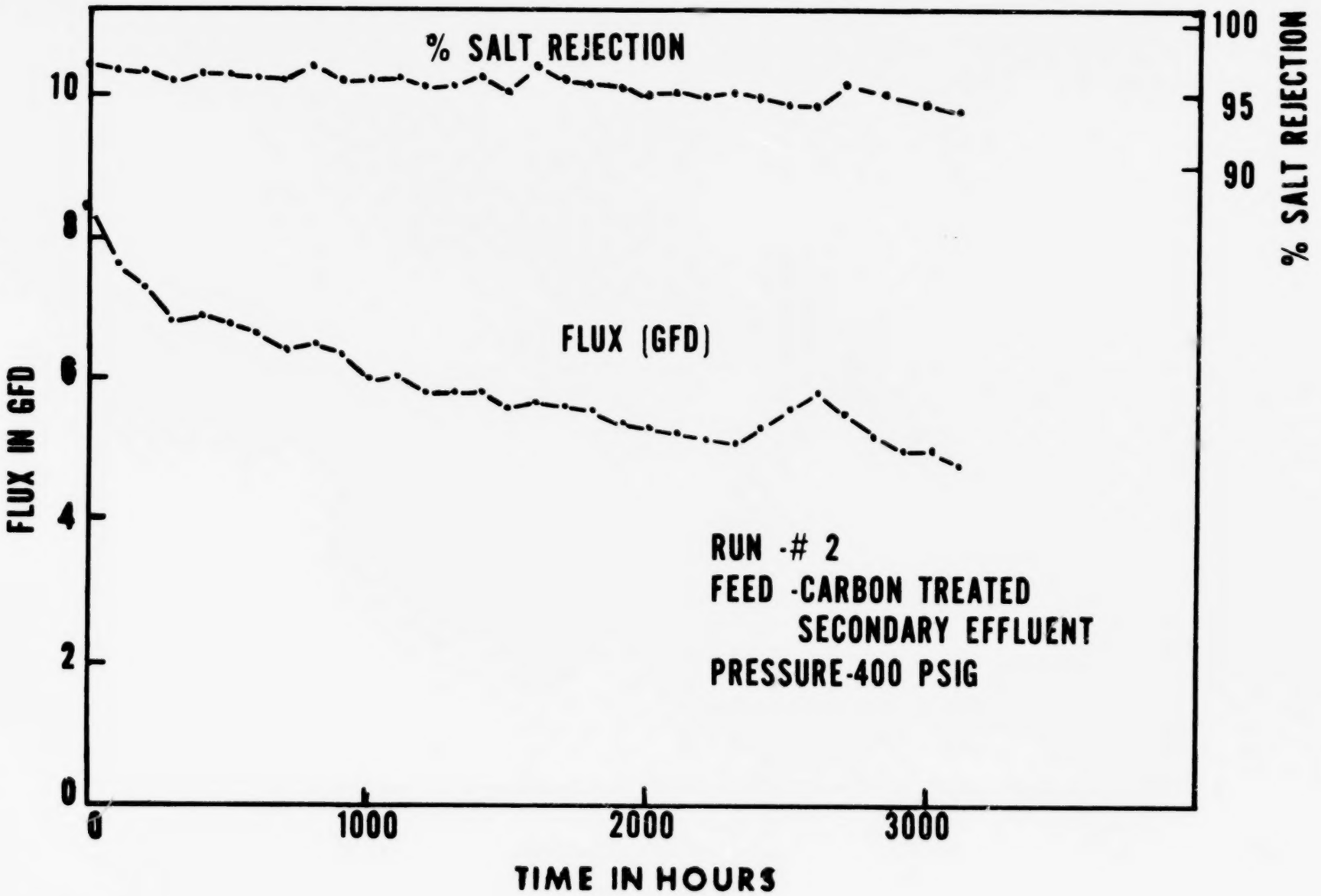


FIGURE 6
FLUX AND SALT REJECTION PERFORMANCE FOR GULF GENERAL
ATOMIC SPIRAL-WOUND REVERSE OSMOSIS MODULE, POMONA, CALIFORNIA

to a replacement of six modules in the unit and partially to slight leakage of feed water through the membrane which developed during that period. The increase in flux rate between 1600 and 1700 hours resulted from an increased leakage of feed water through the membranes which is also reflected in the salt rejection curve of Figure 5.

The only abnormality in the flux vs. time curve for Run #2 occurs between 2300 and 2600 hours. This is a result of two factors, (1) the replacement of six modules in the unit; and (2) a one-week depressurized flushing of the unit with tap water.

A summary of the feed and product water quality data for Runs #1 and #2 is shown in Table 4. The data for Run #1 is divided into two time periods: 0 - 1600 hours, when all of the modules were functioning properly, and 1600 - 2500 hours, when several of the modules had developed leaks. The differences in the relative level of NO_3-N and NH_3-N between the two time periods shown in Table 4 resulted from the method of operation of the Pomona secondary treatment plant. All of the data shown in the two tables is based on average values of 24-hour composite samples taken throughout the time period involved. Samples were collected for analyses twice weekly.

As the product-water flow decreased throughout each run, the water recovery also decreased. However, since the final brine flow rather than the feed flow was being held constant, the decline in water recovery did not accurately reflect the declining product water flow. Thus, the decline in water recovery from the beginning to the end of Run #1 was from 80% to 78%. Comparable values for Run 2 were from 80% to 64%.

Attempts were made throughout both experiments to control the flux decline in the membranes or to restore flux after decline had occurred. Previous experiments at Pomona had indicated that a daily 15-minute air-tap water flush with the system in a depressurized condition was successful in controlling excessive pressure drops across the brine side of the modules. The excessive pressure drops resulted from clogging of the brine passageways by suspended material in the feed water. This procedure was followed throughout both experiments, and while it was again successful in controlling head-loss, it did not appear to have any significant effect on restoring flux rates.

Along with the air-tap water flushing, two other methods of cleaning the membranes were investigated. One procedure, which has been successful in cleaning Gulf General Atomic modules fouled when in use on a brackish water, was a citric acid recirculation through the system. The other cleaning attempt was a procedure employing an anionic detergent solution flush. Neither of these procedures were successful in restoring membrane flux.

TABLE 4

FEED AND PRODUCT WATER QUALITY
DATA FOR EXPERIMENTAL RUNS NO. 1 & 2
IN GULF GENERAL ATOMIC REVERSE OSMOSIS UNIT

Run No.1

TIME 0 - 1600 HRS	Feed	Product	Brine	% Rejection
COD (mg/1)	10.8	1.7	43.8	93.8
NH_3-N (mg/1)	9.2	1.7	49.0	94.2
NO_3-N (mg/1)	2.4	0.8	7.5	84.0
PO_4-P (mg/1)	10.1	0.2	57.7	99.4
TDS (mg/1)	623	73	3402	96.4
TIME 1600 - 2500 HRS				
COD (mg/1)	12.2	1.9	37.2	92.3
NH_3-N (mg/1)	5.3	1.5	25.1	90.1
NO_3-N (mg/1)	12.2	6.0	34.9	74.6
PO_4-P (mg/1)	9.0	1.4	43.2	94.6
TDS (mg/1)	543	145	2738	91.2

Run No. 2

COD (mg/1)	11.4	0.7	37.4	97.1
NH_3-N (mg/1)	17.1	2.9	59.9	92.5
NO_3-N (mg/1)	2.1	0.8	8.9	85.5
PO_4-P (mg/1)	9.7	0.07	39.8	99.7
TDS (mg/1)	552	51	2576	96.7

Notes: (1) All analyses run on 24-hour composite samples

(2) % Rejection calculated using average feed where,

$$\text{Average feed} = \frac{\text{feed + brine}}{2}$$

(3) Figures shown are average values.

Each run was interrupted because of a mechanical problem with the Moyno feed pump. In each instance, the unit was inoperative for 3 to 4 weeks while the pump was being repaired. During this time, tap water was run through the unit at line pressure. When the pump was repaired and the unit was returned to normal operation, most of the modules had developed excessive leaks and were producing water of unacceptable quality. The modules remained in operation for several weeks after the run was resumed to see if the leaks would seal themselves; but, to the contrary, the product water quality continued to deteriorate until the end of each run. Investigation of the membranes indicated that the leakage was caused by a combination of membrane deterioration, and failure of glue and seals in the modules. No satisfactory explanation has yet been advanced for the cause of the membrane deterioration.

The difference in initial flux rate between Run #1 and Run #2 is due mainly to improvements in membrane casting and module fabrication techniques made by Gulf General Atomic in the interim period between the start of each run. However, some of the difference may be due to the higher brine flows used during Run #2. While these higher brine flows did not increase the brine velocity in the downstream modules because of the constant diameter modules used during that run, they did result in an increased brine velocity in the upstream modules. This increased brine velocity may have decreased the boundary layer enough to result in higher flux.

In only one instance during the two runs was the effect of high water recovery on the system evident. At the start of Run #2, the pH of the feed water was maintained at 5.5 rather than 5.0 as had been the case in all previous runs. After 200 hours of operation, the flux of the last set of modules on stream had shown a significantly greater decline than the other modules in the system. A one-hour acid flush of these modules returned the flux to a level consistent with the remaining modules. Calculation of solubility levels, along with consideration of boundary layer factors, indicated that a form of calcium phosphate was possibly precipitating on the membrane surface. After acid flushing, the pH was maintained at 5.0 and no further evidence of excessive flux decline was observed in the last set of modules.

Results of these two experimental runs support the following observations:

1. The extent of flux decline in the spiral-wound reverse osmosis system operating on activated carbon effluent is far greater than would be tolerable in a practical system.

2. The quality of the water produced by the reverse osmosis process is excellent.

3. An air-tap water-flushing procedure was successful in controlling headloss through the system.

4. All attempts to clean fouled membranes were unsuccessful.

5. The high-water recovery obtained in these experiments had no adverse effect on the downstream modules at a pH of 5.0.

6. The cause of the apparent membrane deterioration during prolonged tap-water flushing has not been determined.

Tubular Configuration

The second configuration to be evaluated at the Pomona Water Renovation Plant was of tubular design, manufactured and furnished by the Havens Company.* The module configuration for this unit is shown in Figure 7. A module consists of seven, 8-foot long, 1/2-inch inside diameter fiberglass tubes coated on the inside with a cellulose acetate membrane. The individual tubes are arranged in series and are enclosed in a PVC shroud to collect the product water that permeates from the feed stream side (inside) to the outside of each tube. The complete unit included 48 modules, a high pressure pump, acid addition equipment and the necessary piping, valves and controls.

Six separate experiments were conducted during this phase of the investigation utilizing three membrane types (designated as Type 3A, 4A, and 5A) on two waste streams, secondary effluent and granular carbon-treated secondary effluent. Types 3A, 4A and 5A refer to the relative "tightness" of the membranes. The operating conditions for each of the six experiments are summarized in Table 5. The quality of the feed and product water for both feed streams is tabulated and shown in Table 6. The membrane flux for both clean and fouled modules is compared to the "rated" value in Table 7. A normalized flux decline pattern for both feed streams is shown in Figure 8.

These results demonstrated greater than 92% removal of both organic and inorganic solutes with the exception of NH_3-N and NO_3-N, which were poorly rejected at pH 5.0. Examination of Table 7 illustrates that the "clean module flux" values are much lower than the "rated flux" for the two higher flux modules, and that the flux decline was more severe for the higher flux module, i.e. 35% for the 3A module rated at 27.0 GFD and 10% for the 5A module rated at 10 GFD. Comparison of the flux decline slopes shown in Figure 8 for each feed indicates a two-week flux decline of 13% for the carbon-treated secondary effluent and a 17% decline for the secondary effluent feed. While this difference is not large, it may be attributed to higher COD and turbidity of the secondary effluent.

* Havens Industries, San Diego, California. Mention of products by name does not constitute endorsement by the Federal Water Quality Administration.

FIGURE 7
SCHEMATIC OF HAVENS
REVERSE OSMOSIS MODULE

1) FIBERGLASS TUBE
2) OSMOTIC MEMBRANE
3) END FITTING
4) PVC SHROUD
   to collect product water
5) PRODUCT WATER
6) FEED SOLUTION
7) EFFLUENT

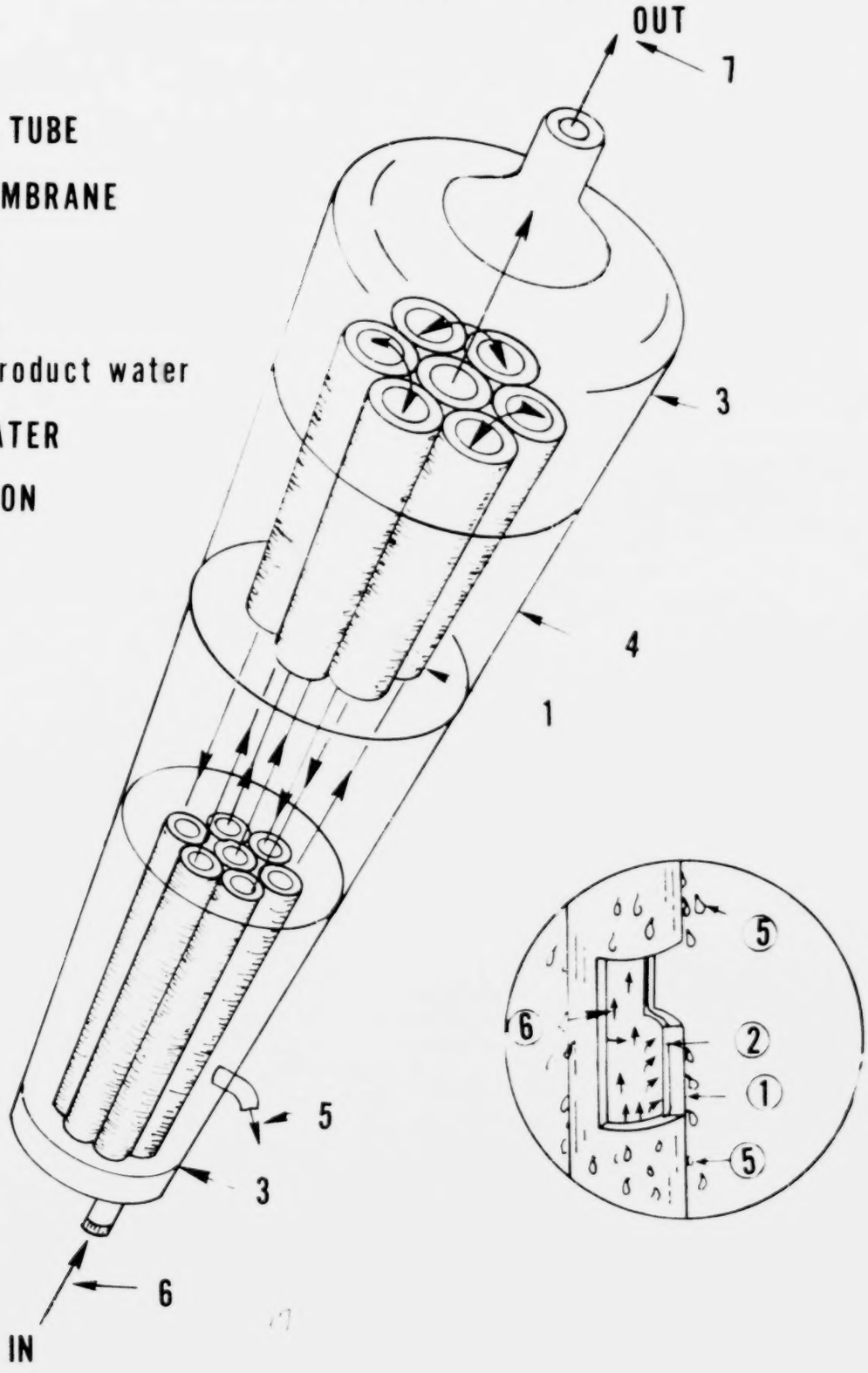


TABLE 5

SUMMARY OF EXPERIMENTS ON HAVENS REVERSE OSMOSIS UNIT

Experiment	Feed	Feed Pressure (psi)	Feed Flow (gpm)	Type Module	Additive To Influent	Duration (hrs)	Initial Recovery (%)	Final Recovery (%)	Reason For Termination
I	Secondary Effluent	800	2.5	4A,5A	50 mg/l Sodium Sulfite	20	78	48	Rapid Flux Decline
II	Secondary Effluent	800	2.5	4A,5A	5 mg/l Calgon CL-77	25	75	45	Rapid Flux Decline
III	Secondary Effluent	700	3.0	3A,4A, 5A	(See Note 1)	325	58	47	Pump Failure
IV	Activated Carbon Effluent	800	3.2	4A,5A	(See Note 1)	140	50	50	Pump Failure
V	Activated Carbon Effluent	850	3.8	4A,5A	(See Note 1)	170	78	78	Excessive Module Breakage
VI	Activated Carbon Effluent	800	3.7	4A,5A	(See Note 1)	460	69	59	Excessive Module Breakage

Notes: (1) 8% Sulfuric acid added to control product water pH at 5.0 ±

(2) No acid addition during first 40 hours.

TABLE 6

WATER QUALITY DATA FOR HAVENS REVERSE OSMOSIS UNIT

Secondary Effluent as Feed	Number of Samples	Feed	Product	% Rejection
COD (mg/l)	8	45.3	1.9	95.8
Turbidity (JTU)	6	12.8	0.1	99.4
TDS (mg/l)	4	607	26	95.7
PO_4 (mg/l)	6	30.5	0.15	99.5
NH_3-N (mg/l)	4	11.4	1.5	86.8
Activated Carbon Effluent As Feed				
COD (mg/l)	7	8.9	0.6	93.4
TDS (mg/l)	5	536	40	92.5
PO_4 (mg/l)	6	26.3	0.25	99.0
NH_3-N (mg/l)	3	10.9	1.4	87.0
NO_3-N (mg/l)	4	3.9	1.2	69.4
Cl (mg/l)	2	121	10	91.9

Note: All analyses run on grab samples.

TABLE 7

COMPARISON OF MODULE FLUX

Module Type	Rated Flux (gpd/ft^2)	"Clean" Modules Flux (gpd/ft^2) Ave.	"Clean" Modules Flux (gpd/ft^2) Range	"Fouled" Modules Flux (gpd/ft^2) Ave.	"Fouled" Modules Flux (gpd/ft^2) Range	% Decline From Clean State
3A	27	17.5	14.0-20.8	11.4	9.1-14.0	35
4A	18	12.4	11.3-14.0	9.7	8.0-12.4	22
5A	10	10.8	8.6-12.0	9.5	8.2-11.5	10

Notes:

1. Rated flux obtained using 5,000 mg/l sodium chloride solution at 600 psi, 25°C, flow rate of 1.5 gpm. Values as reported in Havens Industries literature.

2. "Clean" and "Fouled" fluxes obtained at 800 psi, influent flow rate of 2.5 gpm and influent TDS of 600 mg/l.

3. "Clean" modules flux represents values taken shortly after experiment was begun.

4. "Fouled" modules flux represents values taken after recovery had fallen to 45% - 50%.

5. "Clean" and "Fouled" modules flux represent readings from fourteen Type 3A modules, seven Type 4A modules and ten Type 5A modules.

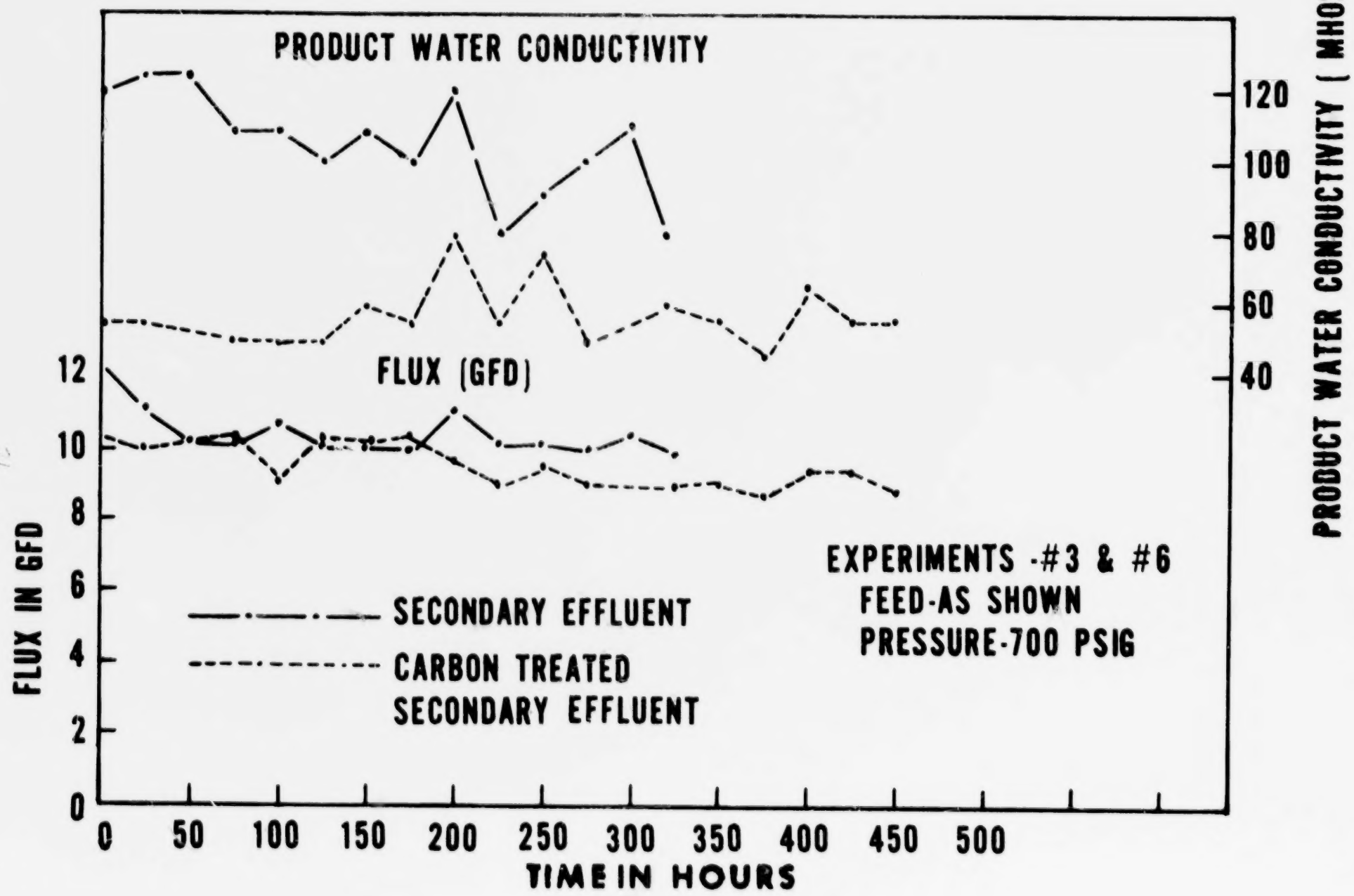


FIGURE 8

FLUX AND PRODUCT WATER CONDUCTIVITY FOR HAVENS TUBULAR REVERSE OSMOSIS UNIT, POMONA , CALIFORNIA

These early results, employing the tubular configuration, were sufficiently encouraging to warrant continuing the investigation. A Universal Water Corporation reverse osmosis unit was used for this second-stage effort. This system utilized a 1/2-inch plastic perforated tube approximately 50 inches long, into which is fitted a preformed tubular membrane sealed at each end with O-rings. Eighteen of these elements were connected in series and placed within a 4-inch diameter PVC pipe to form a single module that contains 7 sq ft of membrane surface. The complete experimental unit was comprised of thirty-two of these modules arranged in banks of four modules each and plumbed originally in a 5-3 array as shown in Figure 9. This arrangement was modified after Run #1 to increase the initial system recovery from 40% to 73%. Two experimental runs have been completed to date with this unit using granular carbon-treated secondary effluent as feed.

Run #1 was conducted at a feed pressure of 600 PSIG with the feed acidified to pH 5.0 using sulfuric acid. A brine flow of 4.90 GPM was maintained with a feed flow of 8.10 GPM and product water flow of 3.20 GPM. The total headloss across the system increased from an original value of 90 PSIG to a final value of 110 PSIG. The flux decline and salt rejection for Run #1 are shown as a function of time in Figure 10, and the feed and product water quality are presented in Table 8. Run #1 was terminated after 1000 hours in order to replumb the unit in a 3-2-2-1 array in order to achieve higher overall recovery. Following the shutdown, the modules were flushed for one hour with a 0.75% bioenzyme solution* that had proven effective in restoring the flux decline in a Gulf General Atomic reverse osmosis unit also being tested at Pomona. This technique increased the flux from 18.0 GFD to 20.6 GFD, compared to an original flux of 23.9 GFD.

Run #2 was conducted under the same conditions as Run #1 except for the recovery ratio, which was increased from 40% to 73%. This caused the exit brine flow to decrease from 4.9 GPM, which for a 1/2-inch diameter tube is equivalent to a bulk velocity of 2.0 ft/sec, to 1.0 GPM which corresponds to a bulk velocity of 0.4 ft/sec. The effect of the decreased velocity and higher recovery can be seen by comparing the flux decline for Run #2 shown in Figure 11, to that exhibited by Run #1 shown in Figure 10. Flux decline for Run #1 was 11% during the first 300 hours compared to 41% during the first 300 hours of the high recovery run. The flux decline was observed to be more pronounced for the downstream modules, confirming the detrimental effects of the poor hydrodynamic conditions experienced by these modules. This condition was probably aggrevated by the increased brine concentration in the later modules. Following the first 300 hours of operation, the unit was depressurized to accommodate regeneration of the granular carbon columns. This caused a significant, but temporary, increase in membrane flux as did an enzyme detergent flush at 450 hours.

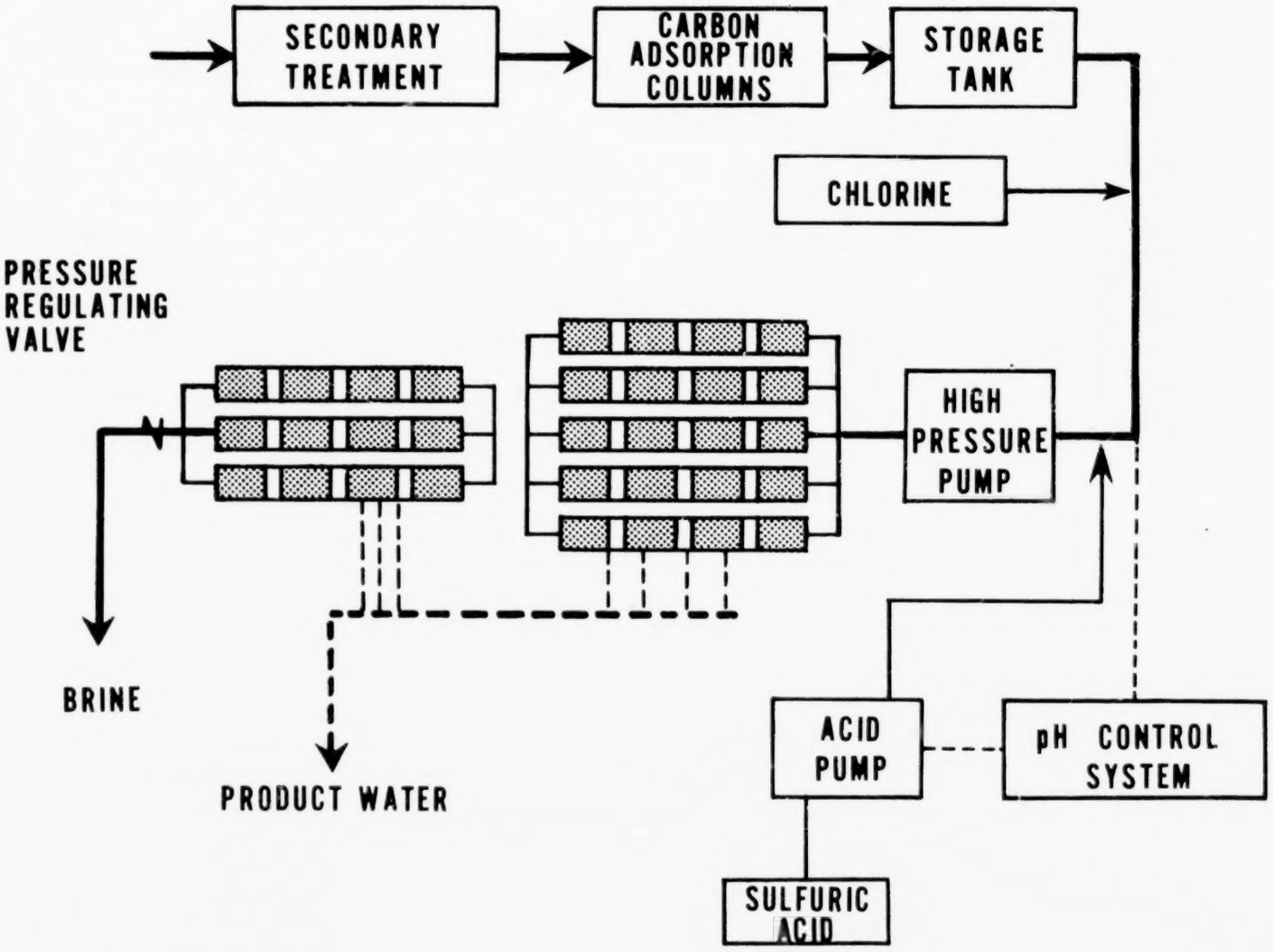


FIGURE 9
FLOW DIAGRAM, UNIVERSAL WATER CORPORATION
TUBULAR REVERSE OSMOSIS UNIT, POMONA, CALIFORNIA

TABLE 8

SUMMARY OF FEED AND PRODUCT WATER QUALITY FOR UNIVERSAL WATER CORPORATION UNIT EXPERIMENTAL RUN NO. 1

Chemical Analysis	No. of Tests	Feed		Product		Brine		Percent Rejection
		Ave.	Range	Ave.	Range	Ave.	Range	
Total COD	10	9.4	7.2 - 12.7	0.3	0.0 - 1.0	14.6	11.1 - 19.0	96.8
Dissolved COD	9	8.3	6.3 - 12.0	-	-	12.7	9.7 - 18.6	-
NO_3-N	9	3.6	0.1 - 12.0	0.7	0.0 - 2.2	5.5	0.1 - 19.0	80.6
NH_3-N	10	11.7	2.5 - 18.1	0.7	0.2 - 1.4	18.9	3.5 - 36.5	94.0
PO_4-P	8	10.1	8.8 - 11.7	0.12	0.0 - 0.4	15.5	12.9 - 18.9	98.8
TDS	10	6.9	5.4 - 711	39	20 - 72	1018	633 - 1859	93.7
Ca	4	39.6		>1		55.4		>97.5
Mg	3	24.5		2.0		39.3		91.8
K	4	12.1		0.8		17.9		93.4
Na	4	111		7.6		156		92.8
SO_4	3	227		0		383		100.0
Cl	3	85		8		153		90.6

Notes: (1) All analyses run on grab samples.
(2) Feed samples taken after acidification with H_2SO_4; hence values of SO_4 shown in table include sulfate contributed by acid addition.
(3) All values shown are in mg/l.

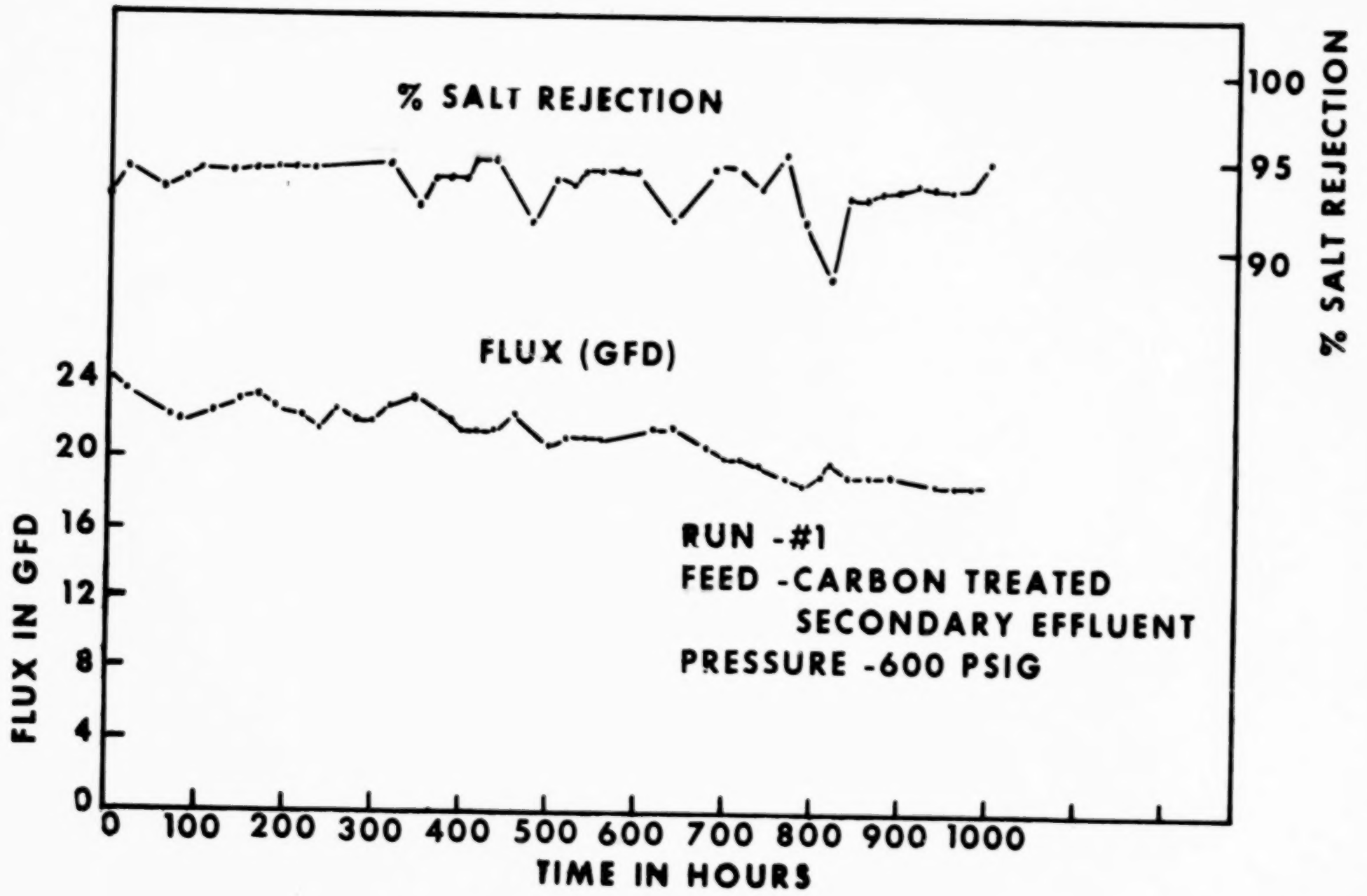


FIGURE 10
FLUX AND SALT REJECTION PERFORMANCE OF UNIVERSAL
WATER CORPORATION TUBULAR UNIT, POMONA, CALIFORNIA

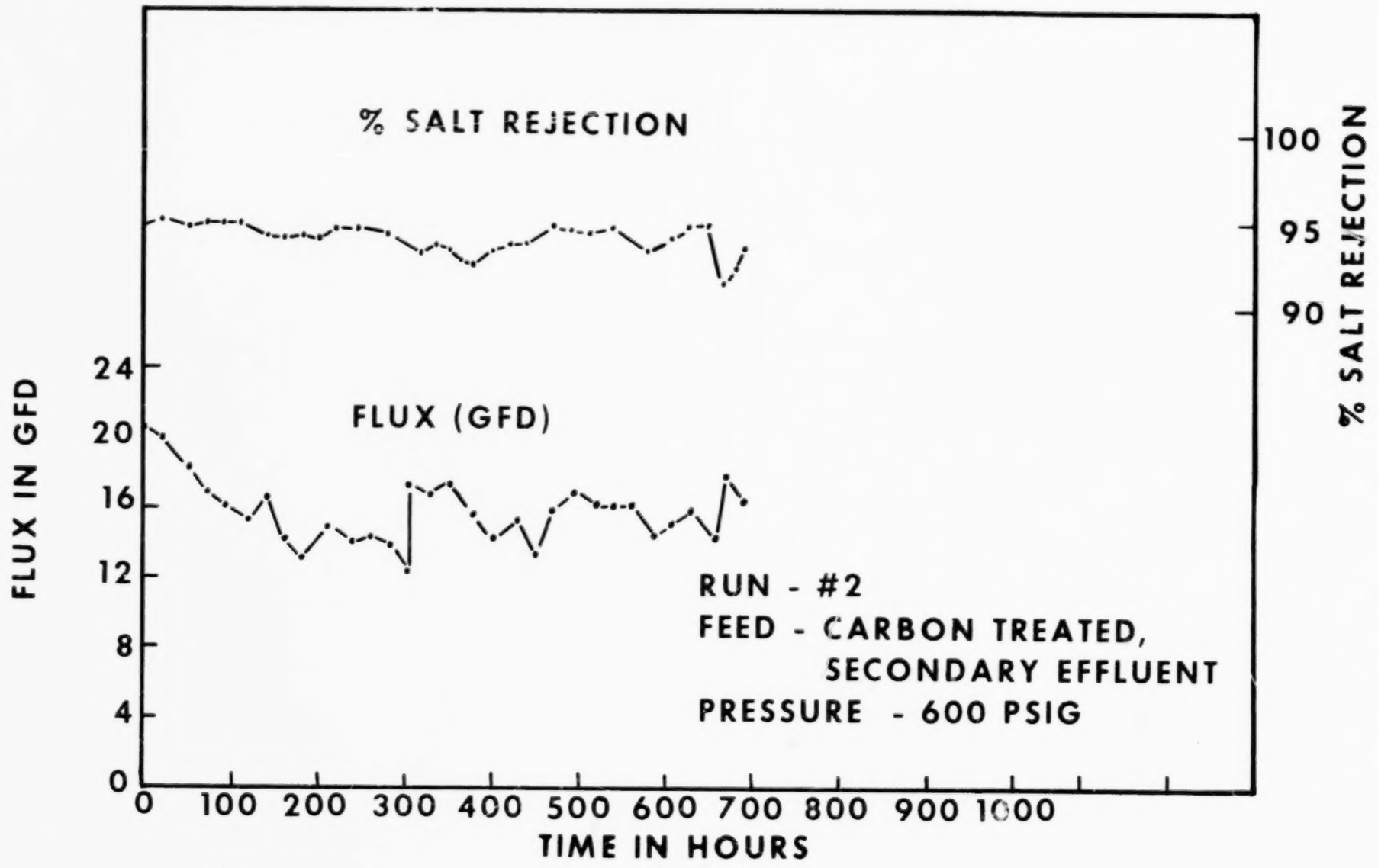


FIGURE 11
FLUX AND SALT REJECTION PERFORMANCE OF UNIVERSAL WATER CORPORATION TUBULAR UNIT, POMONA , CALIFORNIA

(Lebanon, Ohio)

Flat Plate Configuration

Experimental investigation of reverse osmosis at the Lebanon, Ohio Pilot Plant has involved the performance evaluation of a 100 sq ft (2000 GPD) flat-plate reverse osmosis unit on waste streams that represent varying degrees of pretreatment. Principal objectives of this study were to demonstrate the efficacy of treating wastewater with a flat-plate configuration and to evaluate the performance characteristics of the experimental unit as a function of feed water quality.

The five waste streams evaluated during the course of this investigation are shown in Figure 12. The modular arrangement of the reverse osmosis cell and a flow diagram of the entire system are shown in Figures 13 and 14, respectively.

The membrane plates are arranged in eight vertical modules within the pressure vessel. The feed stream enters the uppermost module, and passes in series through each succeeding module leaving the eighth module as the reject stream. The product water flows from each module through separate channels within a central collection tube to allow individual product water quality monitoring from each module. The plates are 14-inches in diameter and consist of two molded phenolic half plates bonded together to form a completed plate which encloses a product water channel. The enclosed channel conducts the product water to the central collection tube. The support substrates and cellulose acetate membranes are mounted on the outside of a completed plate. Adjacent plates are then separated by a polystyrene spiral-flow baffle that directs the feed stream across the surface of the membrane. Construction of the half plates and the spiral-flow baffle are shown in Figures 15 and 16, respectively. Each successive module within the pressure vessel contains fewer plates in order to maintain a relative constant brine velocity as the product water is withdrawn from the feed stream.

Referring to Figure 14, each of the five waste streams evaluated enters an equalization tank containing a thermostatically-controlled immersion heater that acts as a heat exchanger to maintain the feed temperature between 22 and 25°C. After leaving this tank, the pH of the feed is automatically controlled at 5.0 $\pm$ 0.2 units by the automatic addition of sulfuric acid. The five micron filters preceding the high pressure pump were used as a precautionary measure during Runs #1 and #2, but were removed for the remaining three runs. The operating pressure was held within the range 450 to 485 PSIG throughout the entire study.

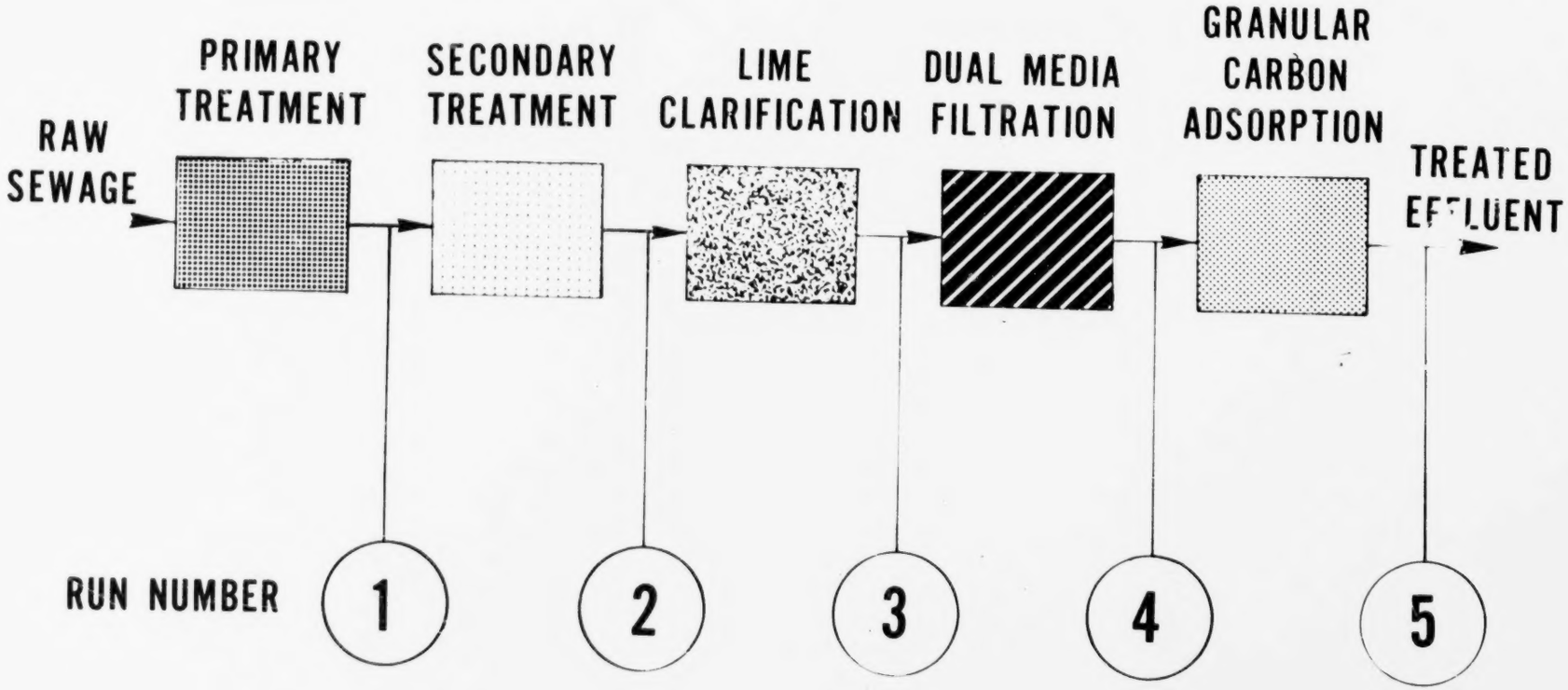


FIGURE 12

SCHEMATIC DIAGRAM OF WASTE STREAMS

LEBANON, OHIO

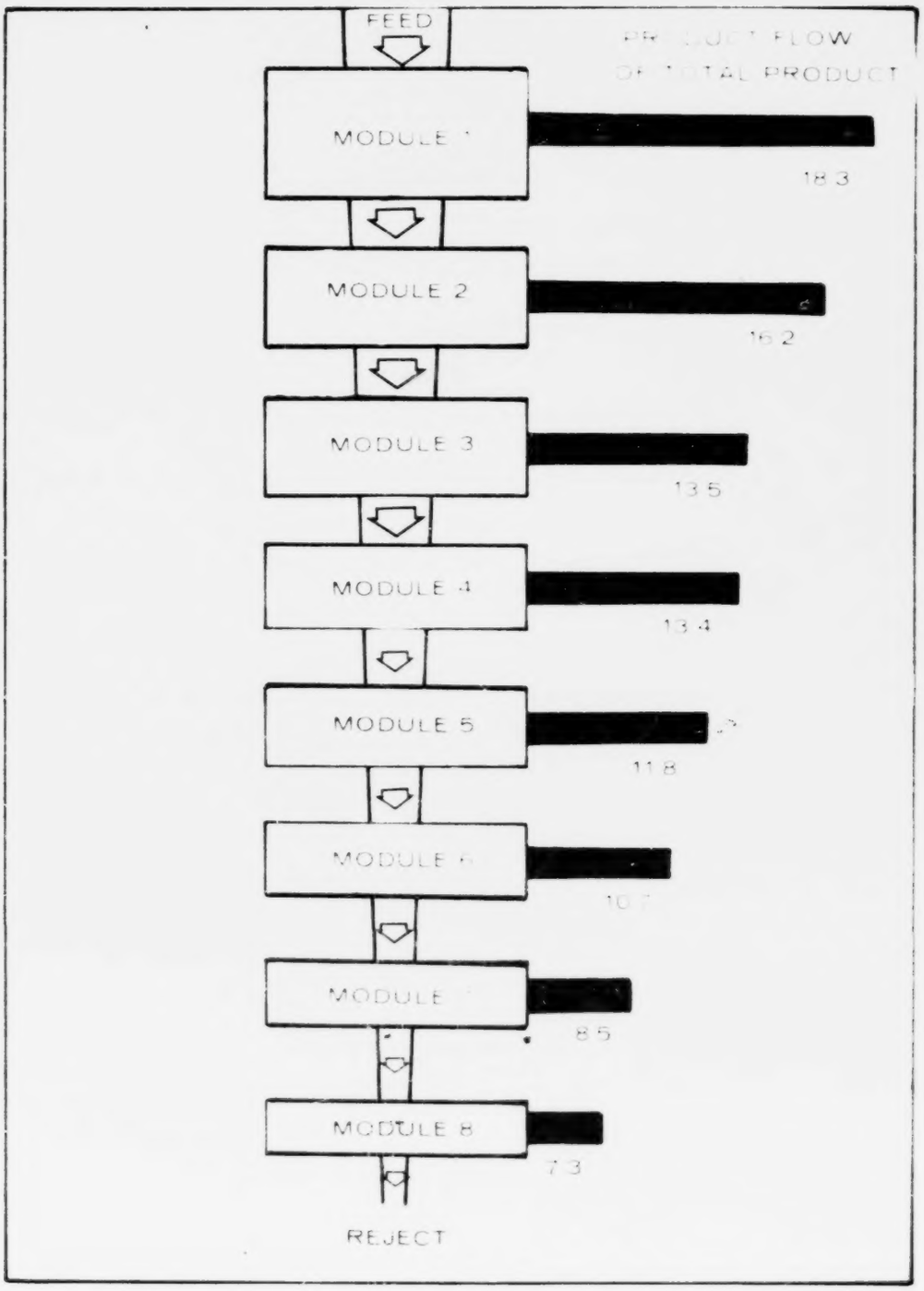


FIGURE 13
MODULAR ARRANGEMENT OF AEROJET GENERAL
FLAT PLATE CONFIGURATION

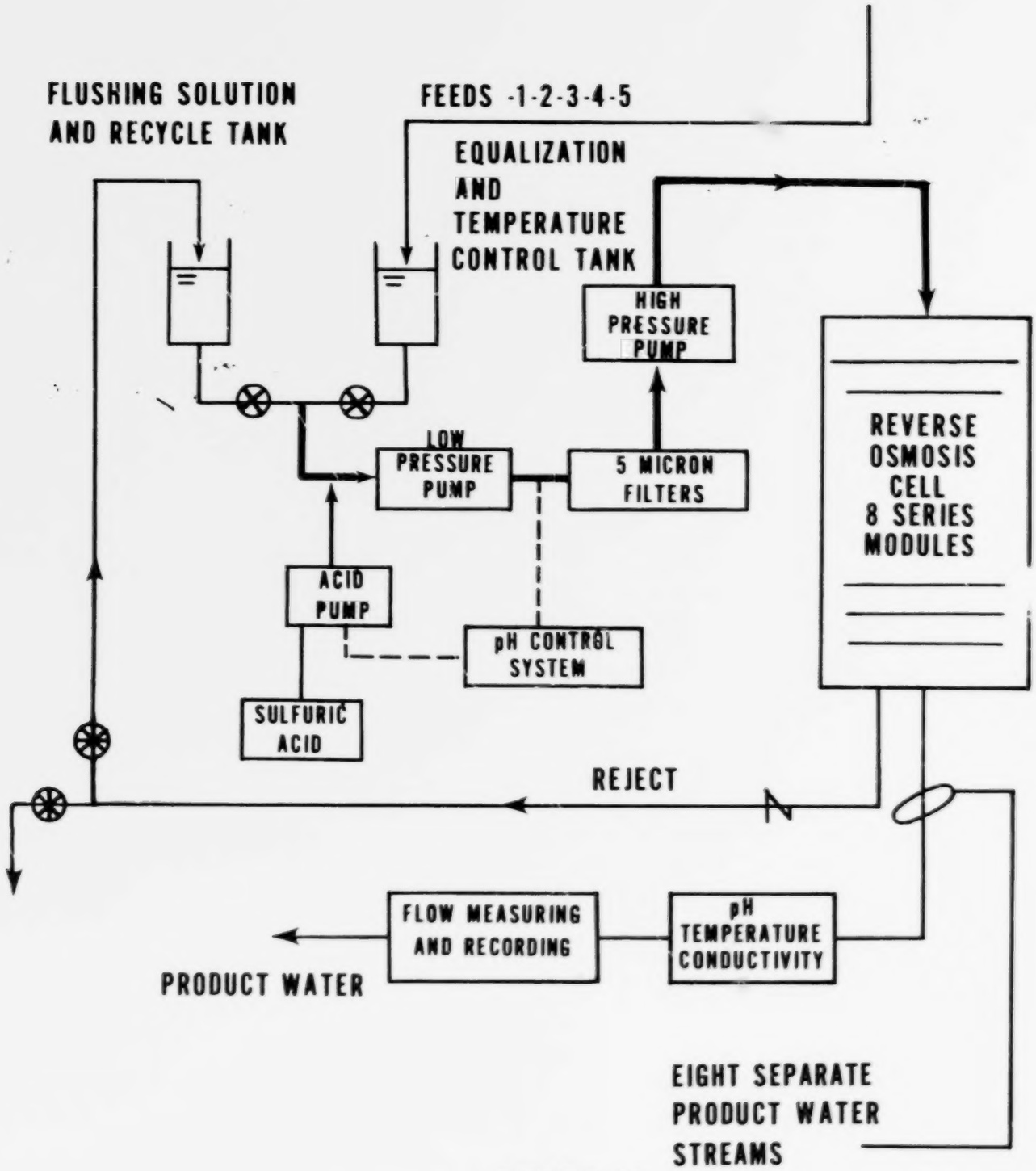


FIGURE 14

FLOW DIAGRAM, AEROJET GENERAL
PLATE & FRAME REVERSE OSMOSIS UNIT, LEBANON ,OHIO

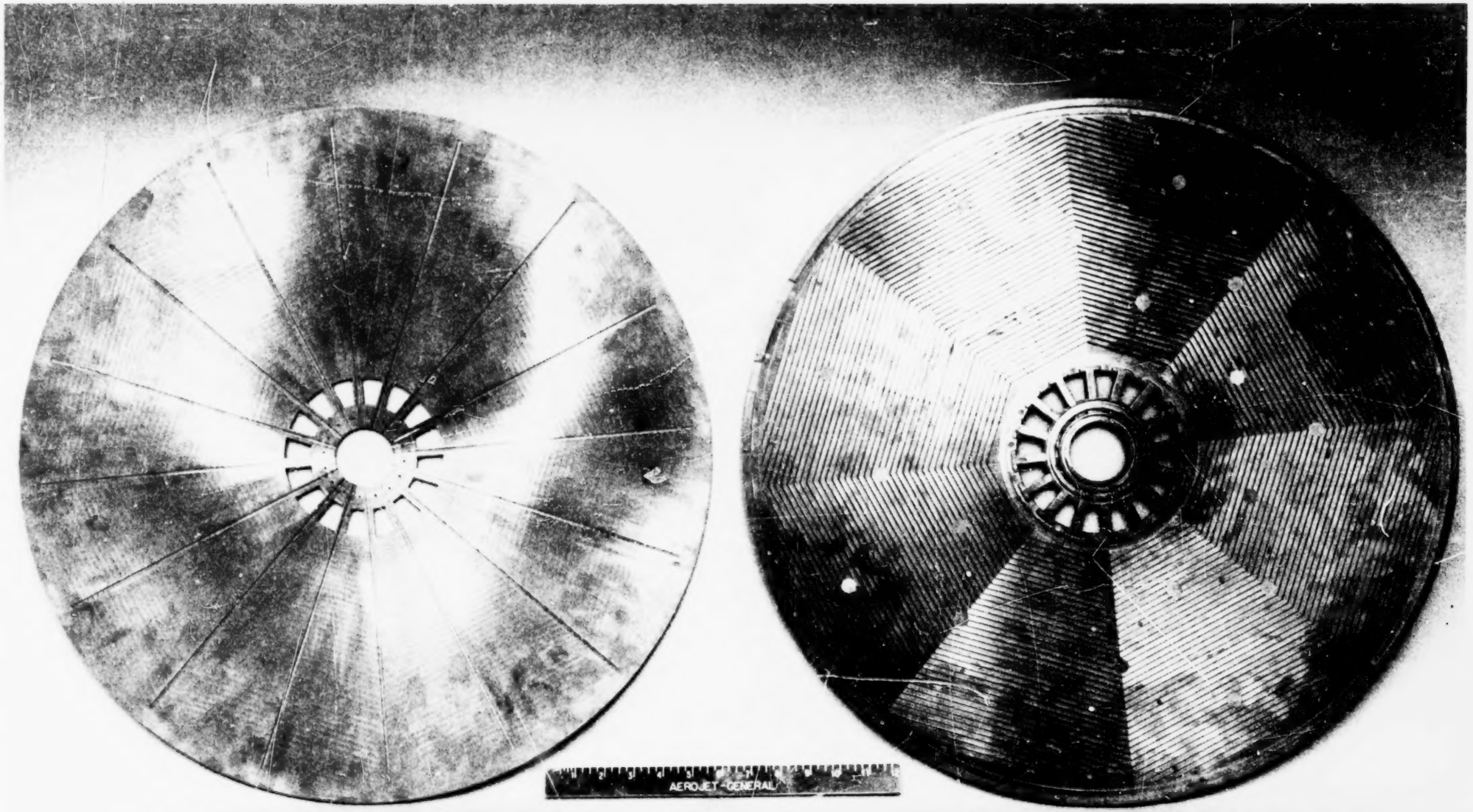


FIGURE 15
TWO PHENOLIC HALF PLATES
14-INCH DIAMETER AEROJET-GENERAL PLAT AND FRAME UNIT

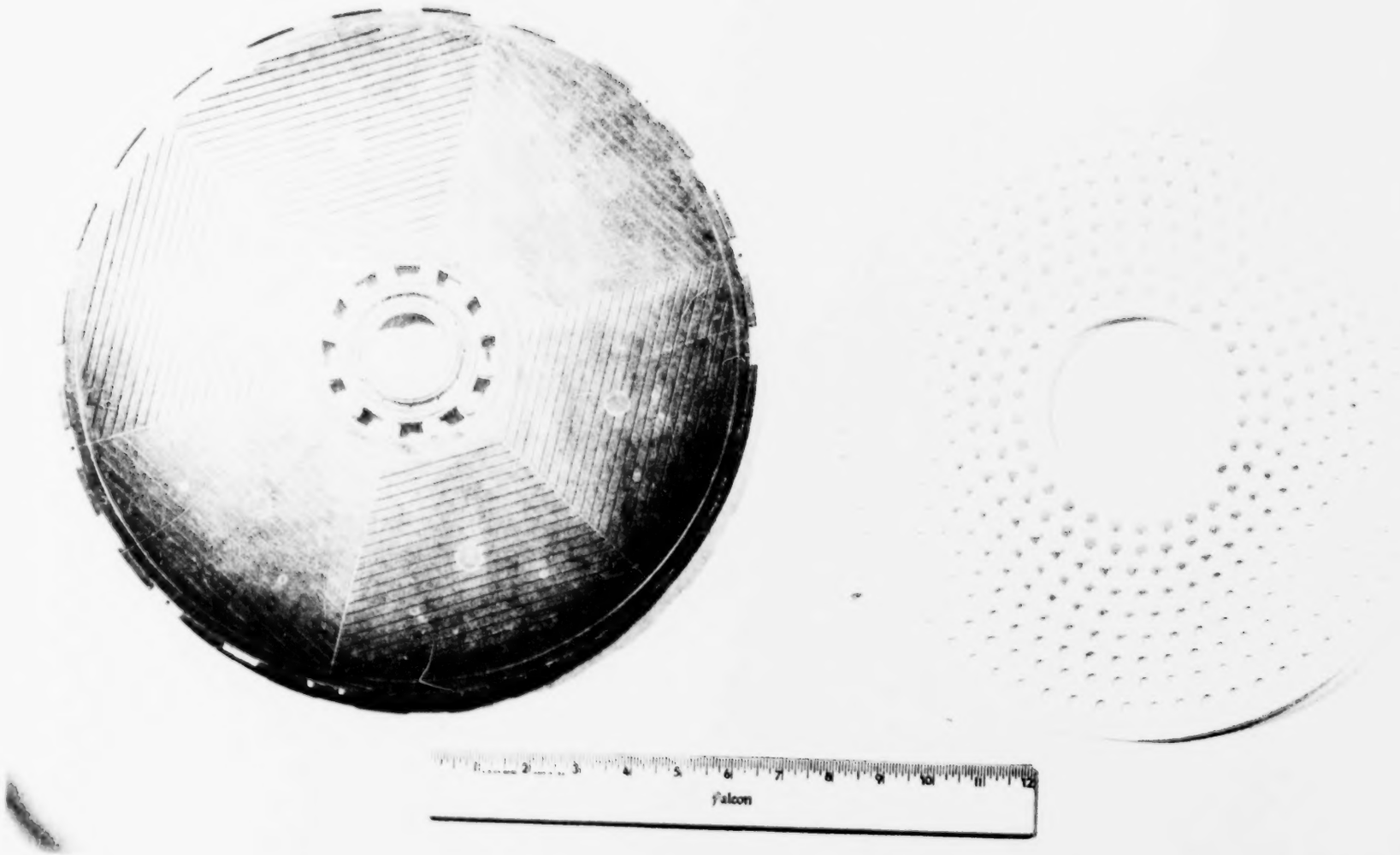


FIGURE 16
PHENOLIC HALF PLATE AND SPIRAL FLOW BAFFEL
14-INCH DIAMETER AEROJET-GENERAL PLATE AND
FRAME UNIT

The feed flow rate to the cell was controlled at 1.8 GPM by a variable speed positive displacement pump. This flow rate resulted in an initial volumetric recovery of 52%, which ultimately declined during the course of the investigation to 35% due to the irreversible membrane fouling that occurred.

The reverse osmosis unit was operated continuously during each run unless interrupted by mechanical difficulties (which were infrequent after the first week of Run #1). The desalination cell was depressurized and stored in a 0.25% formaldehyde solution between runs, or for periods of storage greater than two weeks.

The first experimental run was conducted on lime-clarified, dual-media filtered, and carbon-treated secondary effluent. The flux decline pattern and salt rejection performance for this run are presented in Figure 17 and the rejection efficiency is tabulated and shown in Table 9.

TABLE 9

FEED AND PRODUCT WATER QUALITY DATA
(Run #1)
Lime-Clarified, Filtered, Carbon-Treated
Secondary Effluent

	Feed mg/l	Product mg/l
PO_4 as P	0.60	< 0.1
NO_2-N	0.90	0.60
NO_3-N	3.0	1.1
NH_3-N	< 0.1	< 0.1
Organic-N	< 0.2	< 0.1
TOC	1.0	0.5
Turbidity (JTU)	< 0.5	< 0.1

Results based on average of 10 determinations.

As can be seen from the above table, the feed concentration of the parameters of interest are too low to allow meaningful interpretation of the rejection performance of the membranes. The TDS rejection averaged 90.1% during the run as determined by feed and product water conductivity measurements. The rejection performance of each

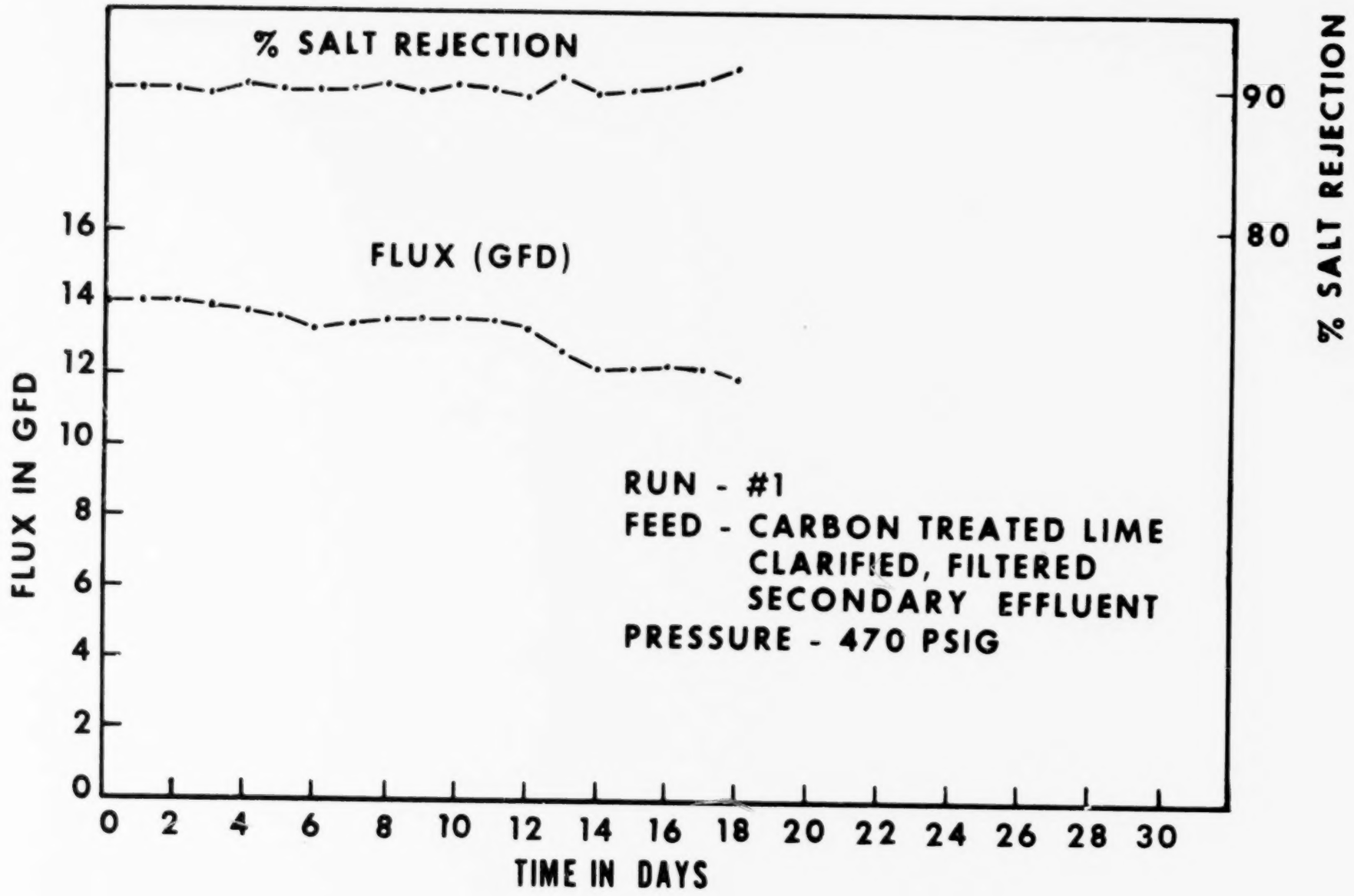


FIGURE 17

FLUX AND SALT REJECTION PERFORMANCE OF AEROJET GENERAL FLAT PLATE CELL, LEBANON, OHIO

module was examined independently during the first run to determine the effect of the increasing brine and organic solute concentration on rejection performance. The results indicated that both the salt and water permeation rates were constant over the entire length of the cell.

The pressure drop across the brine side of the cell was 17 PSIG and remained constant for this run with a feed flow of 1.8 GPM.

The second run was made under the same conditions as Run #1 except that the granular carbon adsorption columns were omitted from the pretreatment train. This run was interrupted on seven separate occasions due to operating schedules, and was finally terminated after 127 days. The flux decline pattern and salt rejection performance are shown in Figure 18, while the feed and product water quality data are shown in Table 10.

TABLE 10

FEED AND PRODUCT WATER QUALITY
(Run #2)

Lime-Clarified, Filtered Secondary Effluent

	Average Values** First 72 days			Average Values*** 72nd to 127th day		
	Feed	Product	% Rejection	Feed	Product	% Rejection
PO_4	1.0	0.1	90	-	-	-
NO_3-N	1.5	0.5	67	2.9	1.2	58
NO_2-N	1.9	1.7	11	*	*	*
NH_3-N	9.6	1.1	88.5	9.5	1.0	89
ORG-N	1.8	0.5	72	2.6	0.4	84.5
TOC	10.6	1.0	90.5	13.3	1.7	95
Total Bacteria Count x 10^6 Per 100 ml	7.3	0.8	99	-	-	-
Turbidity JTU	1.3	< 0.1	-	2.2	< 0.1	-

Results in mg/l unless noted.

* Feed and product NO_2-N less than 0.1 mg/l.

** Average values based on 28 determinations from samples withdrawn at regular intervals during the run.

*** Average values based on 8 determinations from samples withdrawn at regular intervals during the run.

Comparison of the flux decline pattern for the first 8 days of Run #2 to that exhibited by Run #1 demonstrates the effect of the higher organic load as a principal contributor to the increased flux decline. Several efforts to recover this flux decline were made, including daily air and water flushing cycles combined with periodic cell depressurizations as well as attempts to create a "back diffusion potential" to reverse the permeate flow through the membrane. These methods were only marginally successful as shown by Figure 18.

The most promising flux restoration was achieved by flushing the cell for one hour using a 0.7% bioenzyme (BIZ) solution. This procedure resulted in recovery of 63% of the total flux decline experienced during the run. Several attempts to further improve the cleaning efficiency during the flushing cycles were made following this initial success. The most efficient procedure evaluated was to fill the desalination cell with a 0.35% bioenzyme solution at pH 9.7 at a temperature of 25 to 30°C and allow the cell to "soak" for a period of 45 minutes followed by a 15-to 30-minute high velocity flush at an average cell pressure of 50 PSIG. The brine was recirculated without filtration during the flushing cycle which was conducted at twice the normal operating velocity. The TOC and organic nitrogen removals accompanying the first five flushing cycles are given in Table 11.

TABLE 11

REMOVAL OF TOC AND ORGANIC NITROGEN FROM MEMBRANE SURFACE USING A BIOENZYME FLUSHING SOLUTION

Flushing Cycle	Initial Flushing Solution 0.35% Bioenzyme In Product Water		Reject Stream After Completion of 45 min. Soak and 15 min. Flushing Cycle	
	TOC (Total) mg/l	ORG-N mg/l	TOC (Total) mg/l	ORG-N mg/l
1	62	1.1	500	80
2	44	0.4	450	71
3	67	1.1	480	25
4	69	2.1	435	44
5	93	4.0	650	94

The appearance of the flushing stream changes from a light blue color before flushing to a light brown turbid solution following the flushing cycle. Each flushing requires 20 gallons of water, ten of which are contained in the cell, with the remainder used to fill the

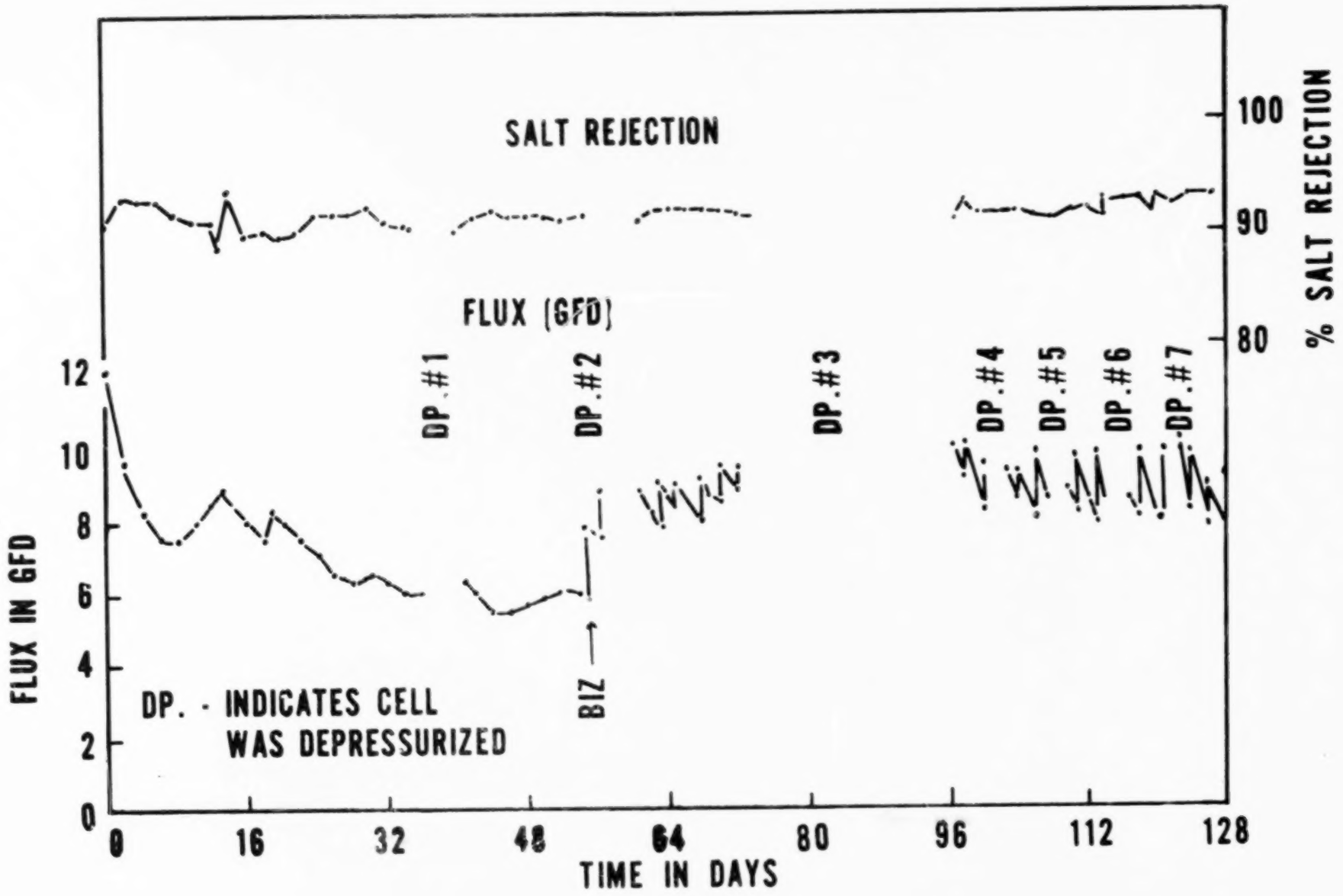


FIGURE 18
FLUX AND SALT REJECTION PERFORMANCE OF AEROJET GENERAL FLAT PLATE CELL, LEBANON, OHIO

recirculating lines, pump and mixing tank. The TOC and organic nitrogen levels shown in Table 11 are representative of the increase that occurs during each cleaning cycle and illustrate the organic nature of the membrane deposit.

The TDS rejection capacity of the membranes remained relatively constant at 92.1% during the run. The repeated exposures of the membranes to a pH of 9.5 during the cleaning procedures have shown no adverse effect on membrane performance thus far. Similarly, there has been no evidence of biological degradation of the membranes.

The third run with the Aerojet-General reverse osmosis unit was conducted on lime-clarified secondary effluent. System operating pressure was maintained at 470 PSIG, with feed stream pH controlled at 5.0 $\pm$ 0.2 units by the addition of sulfuric acid. The feed and product water quality data for this run are shown in Table 12.

TABLE 12

FEED AND PRODUCT WATER QUALITY (RUN #3)

Lime-Clarified Secondary Effluent

	Feed	Product			
		High	Average	Low	Ave.% Rejection
PO_4 as P	2.0	0.1	0.1	0.1	95
NO_2-N	< 0.2	0.4	< 0.2	< 0.1	*
NO_3-N	0.2	0.4	0.1	< 0.1	*
NH_3-N	11.0	1.7	1.1	0.6	90
ORG-N	3.0	1.6	0.7	0.1	86
TOC	20.8	9.0	2.8	0.8	87
Turbidity (JTU)	7.5		< 0.1		99

Results in mg/l unless noted.

* Feed concentrations of NO_2-N and NO_3-N are too low for meaningful rejection data.

Membrane performance was satisfactory throughout this run with TDS and TOC rejection averaging 92% and 87%, respectively. The TDS rejection and flux decline pattern are shown in Figure 19.

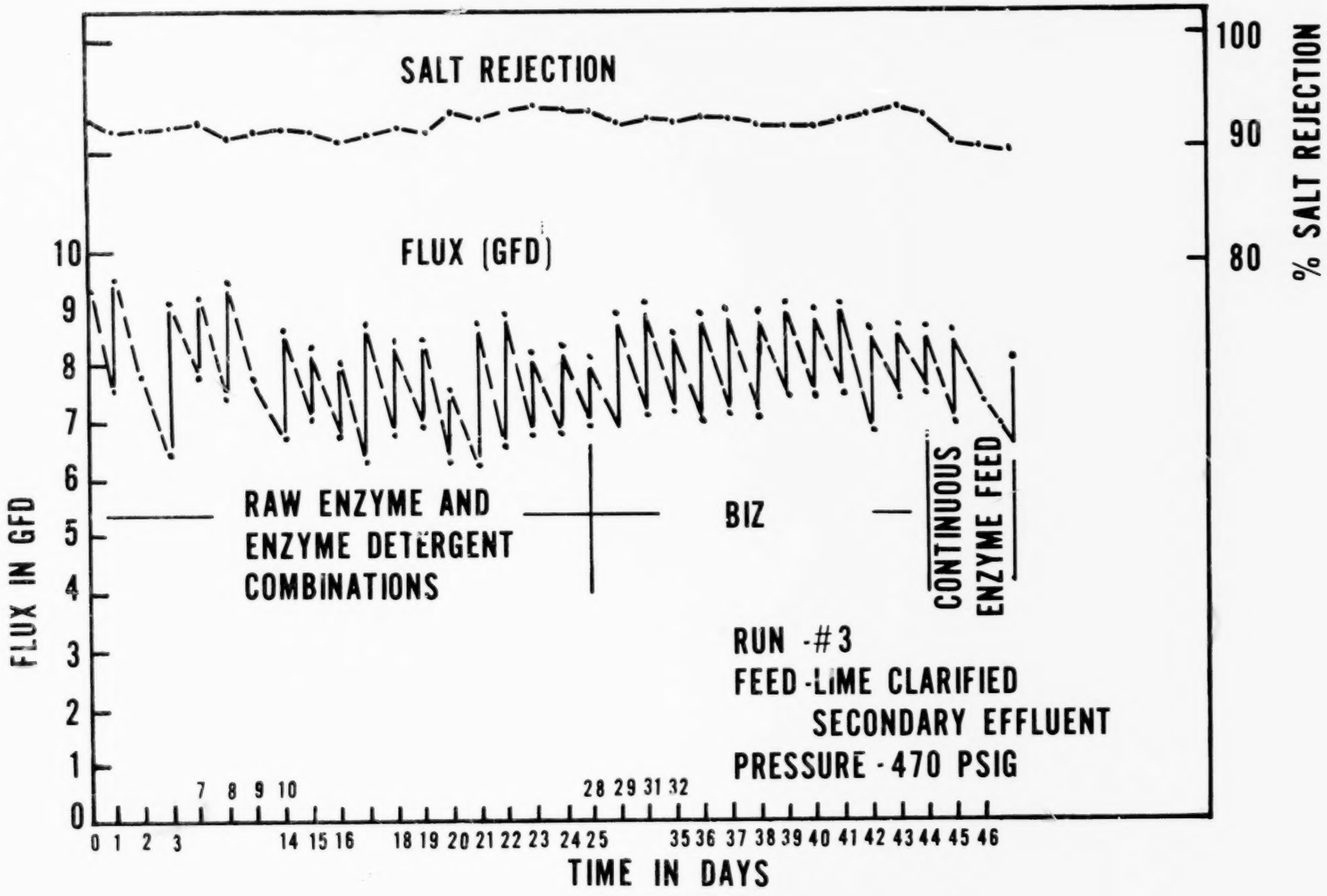


FIGURE 19

FLUX AND SALT REJECTION OF AEROJET GENERAL FLAT PLATE CELL, LEBANON, OHIO

Several standardized flushing cycles were employed during this run using BIZ, as well as a number of raw enzyme detergent combinations. The raw enzymes used were the protease-amylase enzymes manufactured specifically for the detergent industry. A comparison of the relative effectiveness of these enzymes is presented in Table 13. Although results of these flushing procedures are not quantitative, some generalities concerning the effectiveness of the enzymes can be seen.

The raw enzymes appear to be more effective when used with a detergent. Enzyme concentrations less than 35 mg/l and detergent concentrations less than 3500 mg/l tend to limit the cleaning effectiveness for a service interval of 24 hours. The three enzymes evaluated seem to be equally effective in removing the organic fouling layer. An enzyme/detergent ratio of 1 to 100 appears to be the most effective combination evaluated. This ratio approximates the reported concentration of enzyme contained in the BIZ formulation. The effect of pH has not been well defined by the evaluation but information from enzyme suppliers indicates that the optimum pH for the detergent enzymes is between 7.5 and 9.5.

The percent flux increase associated with each flushing cycle is highly variable, depending on the amount and nature of the organic material deposited during the previous service cycle. A portion of this fouling layer seems relatively easy to remove as evidenced by the flux increase (6.3 to 7.4 GFD) resulting from the flush with a detergent solution on March 3, 1969. Following completion of this flushing cycle, a BIZ solution resulted in an additional flux recovery (from 7.4 to 8.7 GFD).

The average flux during this run was 7.6 GFD compared to 8.7 GFD for Run #2. This decrease was apparently due to both the higher organic and suspended solids load of the feed and the less efficient flushing cycles employed while comparing the raw enzymes.

The pressure drop across the brine side of the membrane increased from an average value of 20 PSIG for Run #2 to 28 PSIG for the present run. This increased pressure drop is a result of the higher suspended solids concentration of the feed stream which tends to accumulate in the feed channels. Continued accumulation of these solids is easily controlled, however, by the higher velocities associated with the flushing cycles.

The fourth run with the Aerojet-General flat-plate configuration was conducted on secondary effluent. This run, lasting 22 days, was interrupted after the 11th day for a period of 60 days and was then resumed for an additional eleven days. The system pressure averaged 485 PSIG with the feed stream pH controlled at 5.0 $\pm$ 0.2 units during the run. The desalination cell was stored in a 0.25% formaldehyde solution during the 60-day shutdown to prevent possible biological hydrolysis of the membranes.

TABLE 13

EVALUATION OF ENZYME FLUSHING SOLUTIONS

Date	Flushing Solution	Service Preceding Flush in HRS.	Concentration of Flushing Solution in PPM	pH of Flushing Solution	Flux in GFD Before-After	% Flux Increase	Membrane Rejection Performance
2/11/69	BIZ	24	3500	9.5	7.6 - 9.6	26.3	Good
2/13/69	BIZ	48	3500	9.5	6.5 - 9.1	42.0	Good
2/14/69	BIZ	24	3500	9.4	7.8 - 9.1	16.3	Good
2/18/69	HT-Proteolytic	24	3.5	5.2	7.4 - 8.4	13.5	Good
2/20/69	HT-Proteolytic	48	10	5.2	6.8 - 8.3	22.0	Good
2/25/69	HT-Proteolytic	24	10	9.4	6.8 - 8.2	20.6	Good
2/26/69	HT-Proteolytic	24	35	7.0	6.8 - 8.0	17.5	
	with TIDE		10	-			
2/27/69	HT-Proteolytic	24	35	8.8	6.0 - 8.4	40.0	
	with TIDE		3500	-			Good
2/28/69	Milezyme with TIDE	24	35	8.8	6.8 - 8.4	23.6	Good
3/1/69	Milezyme with TIDE	24	35 500	8.6	6.8 - 8.4	23.6	Good
3/2/69	Milezyme	24	10 500	8.6	6.5 - 7.6	17.0	Good
3/3/69	TIDE	24	3500	8.8	6.3 - 7.4	17.5	Good
3/3/69	BIZ	0	3500	9.4	7.4 - 8.7	17.5	Good
3/4/69	BIZ	24	3500	9.5	6.6 - 8.9	35.0	Good
3/5/69	WT-2	24	35	8.8	6.8 - 8.3	22.0	Good
	with TIDE	24	3500				

The rejection performance of the cell is shown in Table 14 for both the period preceding and following the shutdown. In general, rejection of the divalent ions was greater than 94% while the rejection of the monovalent ions was varied from 80 to 90%.

The effect of the 60-day storage on membrane performance is somewhat anomalous. The TDS rejection declined from 91.5% before storage to 86% after storage, a drop of 5.5% as measured directly (by evaporation of composited feed and product water samples). Conductivity measurements show this decline to be 9.3%, from 90.3% before storage, to 81% after storage. This apparent discrepancy can be resolved by considering the increasing percent rejection that occurred following the storage period as shown by Figure 20, and by noting that the TDS samples were composited during the 20th and 23rd day. Conductivity measurements on the product water from the individual modules indicated the decrease in membrane performance was a result of a generalized condition, exhibited by all eight modules rather than a localized condition. The increase in rejection performance with time may result from the "healing" of imperfections present in the membrane. This rationale presumes that previously deposited organic materials were leached out during the storage period, thereby causing the increase in salt permeation.

Organic rejection, as measured by TOC, increased slightly from 89% to 94% during the storage period. No particular significance can be attached to this increase, however, in view of the relatively low TOC levels involved.

The flux decline characteristics for this run are shown in Figure 20, and indicate that a moderate flux decline can be maintained by employing a one-hour daily enzyme flush.

It should be noted that the quality of the secondary effluent was exceptionally good during this run with TOC averaging 14.9 mg/l for the first 11 days and 8.3 mg/l for the remaining 11 days. Feed turbidity similarly decreased from an average of 6.1 JTU during the first half of the run to 2.4 JTU during the last half of the run.

The fifth run was made on primary effluent. This run was conducted at an average operating pressure of 480 PSIG with a feed stream pH of 5.0 $\pm$ 0.2 units. The flux decline characteristics and rejection performance for this run are shown in Figure 21. The flux declined from a daily average of 5.4 GFD on the third day of operation to 3.8 GFD on the 12th day. This represents a 30% decline in flux that occurred in nine days with daily bioenzyme flushing. The 5.4 GFD is significantly lower than the daily average of 7.8 GFD that was maintained during the run on secondary effluent. The feed and product water quality data for this run are presented in Table 15. The TDS rejection decreased from a high of 90% to a low of 84% during this run. This decline in rejection is not surprising in view of the lower flux and poor distribution of the feed caused by plugging of the feed channels.

TABLE 14

FEED AND PRODUCT WATER QUALITY
(Run #4)
Secondary Effluent

Constituent	Before 60-day Shutdown			After 60-day Shutdown			
	Conc. in mg/l*		% Rejection	Conc. in mg/l*		% Rejection	% Change
	Feed	Product		Feed	Product		
Calcium (Ca)	104	5.2	95	110	2	98	+3
Magnesium (Mg)	30.5	0.4	99	28.6	.5	98	-1
Sodium (Na)	95.5	15.0	84	91.0	14.0	85	+1
Potassium (K)	8.5	1.5	82	8.2	1.3	84	+2
Iron (Fe)	<0.3	<0.3	-	0.3	0.1	-	-
Manganese (Mn)	<0.1	<0.1	-	-	-	-	-
Phosphate (PO_4asP)	6.6	0.4	94	6.8	0.6	91	-3
Sulfate (SO_4)	113	14	88 (1)	107	7	93	+5
Chloride (Cl)	133	26	81	131	26	80	-1
Silica (SiO_2)	12.4	4.1	67	13.8	3.4	75	+8
Nitrite (NO_2^-)	<0.2	<0.2	-	-	-	-	-
Nitrate (NO_3^-)	<0.2	<0.2	-	8.6	4.7	45	-
NH_3-N	8.7	1.7	80	1.0	0.3	-	-
Organic-N	2.1	0.3	86	0.9	0.1	89	+3
Total Alk. ($CaCO_3$)	332	39	88	326	40	88	0
TDS (Conductivity	-	-	90.3	-	-	81	-9.3
TDS (Evaporation)	726	66	91.5	644	88	86	-5.5
TOC (Unfiltered)	14.9	1.7	89	8.3	0.5	94	+5
Turbidity (JTU)	6.1	<0.1	-	2.4	<0.1	-	-

* Average of two 24-hour composite samples.

(1) Feed water sample taken before addition of H_2SO_4.

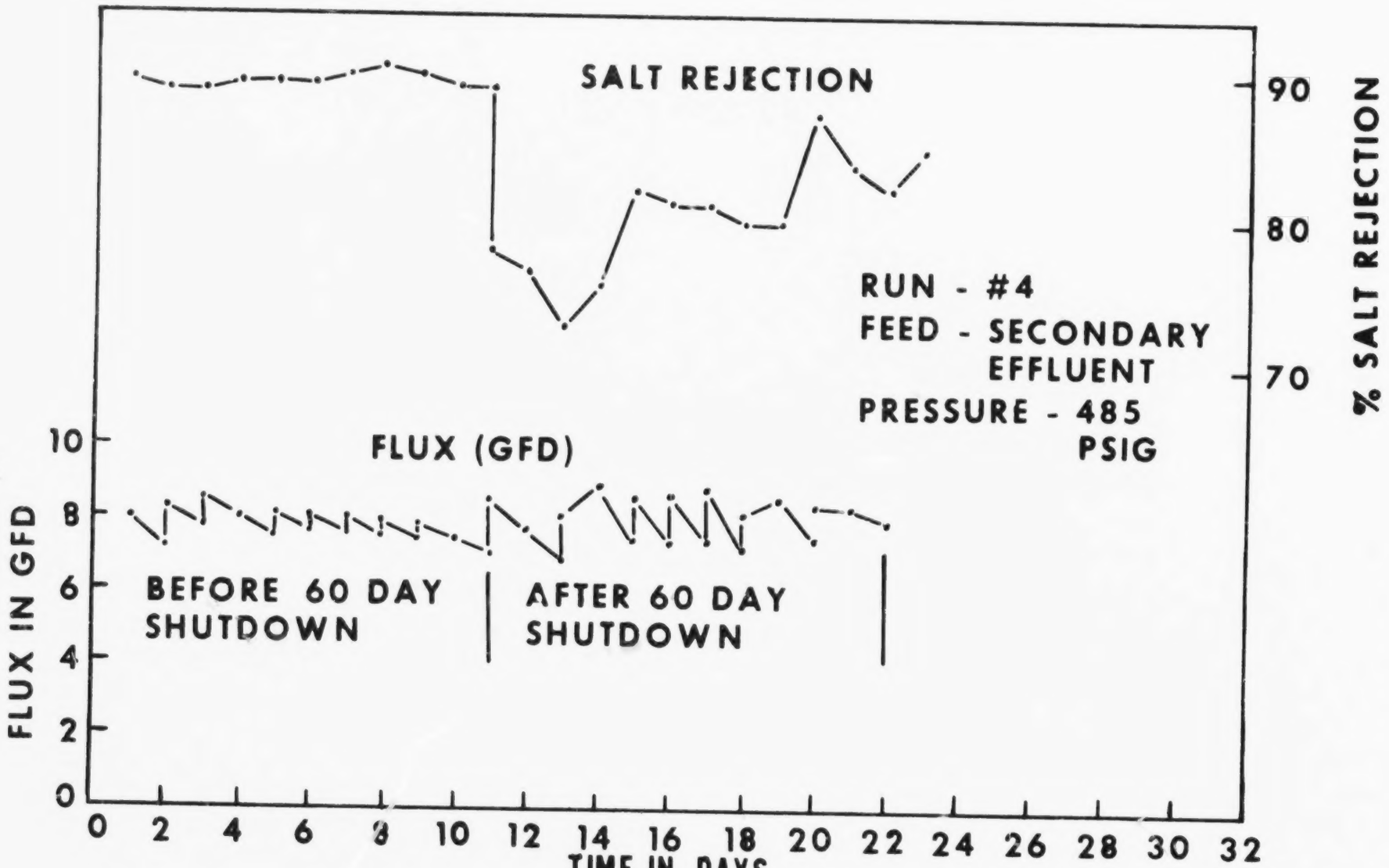


FIGURE 20
FLUX AND SALT REJECTION PERFORMANCE
OF AEROJET GENERAL FLAT PLATE CELL, LEBANON, OHIO

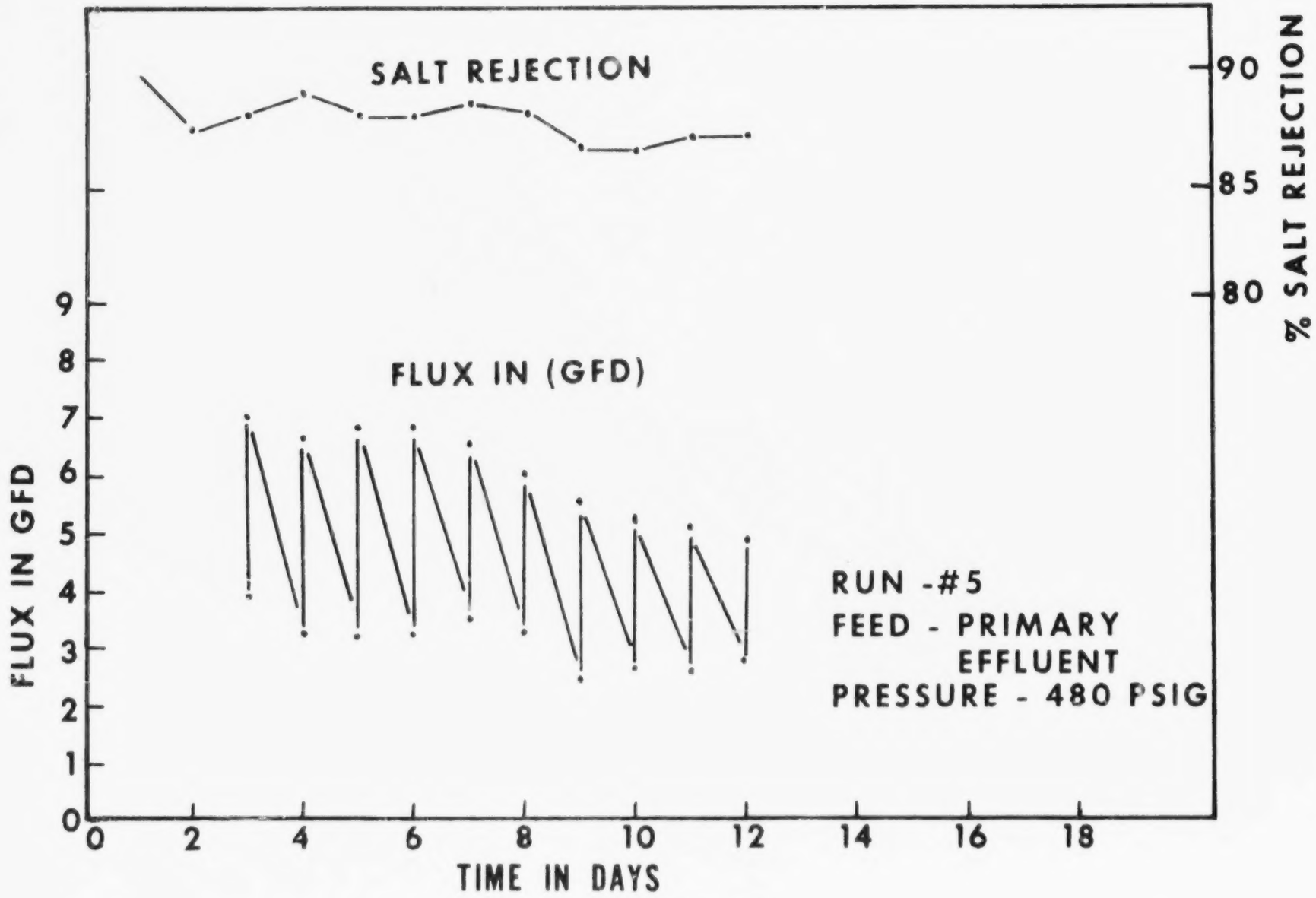


FIGURE 21
FLUX AND SALT REJECTION PERFORMANCE OF AEROJET GENERAL FLAT PLATE CELL, LEBANON, OHIO

TABLE 15

FEED & PRODUCT WATER QUALITY
(Run #5)

Primary Effluent

Constituent	Concentration mg/l		
	Feed mg/l	Product mg/l	% Rejection
Calcium (Ca)	138	2.0	99
Magnesium (Mg)	30.7	0.4	99
Sodium (Na)	141	24.7	82
Potassium (K)	11.0	2.6	76
Iron (Fe)	1.5	0.1	93
Manganese (MN)	*	*	-
Phosphate (PO_4 as P)	9.7	0.4	96
Sulfate (SO_4)	173	5	97 (1)
Chloride (Cl)	168	47	72
Silica (SiO_2)	26.7	6.0	80
Nitrate (NO_3)	*	*	-
Nitrite (NO_3)	*	*	-
NH_3-N	12.5	2.1	83
Organic-N	6.4	0.6	91
Total Alkalinity (as $CaCO_3$)	395	23	94
TDS (Conductivity)	-	-	86
TDS (Evaporation)	917	128	86
TOC (Unfiltered)	97	13.4	89
TOC (Filtered)	35.0	12.0	66
Turbidity (JTU)	52.0	< 0.1	-

* Concentration < 0.1 mg/l.

(1) Feed water sample taken before addition of H_2SO_4.

(2) Feed filtered through glass-fiber filter paper, Type 1106 BH, Mine Safety Appliances Company, Pittsburgh, Pennsylvania.

The most notable change in operating conditions during this run was the more pronounced pressure drop that was experienced across the desalination cell. This drop ranged from a low of 40 PSIG after the daily flushing to over 80 PSIG near the end of the daily service cycle. In many cases it was necessary to decrease the velocity of the feed stream to maintain the pressure drop across the cell to values less than 100 PSIG. This lower brine velocity may have significantly affected the flux decline rate resulting in a distorted comparison to the performance characteristics exhibited by the previous feed streams.

A comparison of the rejection of principal constituents for the five runs conducted at Lebanon is shown in Table 16. These results indicate that the rejection performance was similar for the waste streams evaluated.

TABLE 16

SUMMARY OF REJECTION PERFORMANCE
FOR
LEBANON WASTE STREAMS

Constituent	Run #1	Run #2	Run #3	Run #4	Run #5
PO_4 as P	*	91	90	93	96
NO_3-N	*	68	63	*	*
NH_3-N	*	88	89	80	83
Organic-N	*	89	78	87	91
TOC	*	85	89	94	89
TDS	90	92	92	91**	90
Turbidity(JTU)	*	99	99	99	99

* Feed concentration too low for meaningful rejection data.

** TDS rejection before 60-day storage period.

Experience has shown that the time-dependent flux decline pattern of a membrane system operated on a feed containing both organic and inorganic solutes at constant pressures can be linearized by plotting log flux vs log time; the slope of the resulting straight line of best fit is commonly referred to as the "flux decline slope" and is used to quantitatively describe the change in flux with time.

The flux decline slopes for Runs #1 and #2 are compared in Figure 22. The differences in the flux decline slopes for these two runs indicate the effect of carbon adsorbable material on flux decline. It should be remembered that the feed for both Runs #1 and #2 was passed through a 5 micron cartridge filter prior to entering the desalination cell. This, combined with the increased feed TOC exhibited by Run #2, supports a positive role for soluble and colloidal organic carbon in causing flux decline.

A similar comparison of flux decline slopes for Runs #3, #4, and #5 is presented in Figure 23. These runs were made after removal of the 5 micron filters and reflect the effect of the particulate material in the respective waste streams. It is encouraging to note that, by employing a one-hour bi-daily cleaning schedule for Run #3 - lime-clarified effluent, and a daily cleaning schedule for Run #4 - secondary effluent, respective flux decline slopes of -0.03 and -0.04 could be maintained. These slopes are less than the slope of -0.05 experienced when operating on carbon column effluent, a much higher quality water. These data support the concept of controlling membrane fouling by employing periodic cleaning procedures as an alternate to pretreating the feed to prevent fouling.

The importance of maintaining a reasonable flux decline slope and membrane lifetime can be seen from an examination of Table 17.

TABLE 17

FLUX DECLINE SLOPE LOG FLUX VS LOG TIME	ACCUMULATIVE % LOSS OF ORIGINAL FLUX AFTER YEARS		
	(1)	(2)	(3)
-0.01	5	5.5	6
-0.05	25	28	30
-0.10	44	47	51

The flux decline characteristics shown above indicate that the first-year flux decline represents well over 80% of the decline that may be expected for any reasonable membrane lifetime and that the one-year flux value would provide a rational design figure.

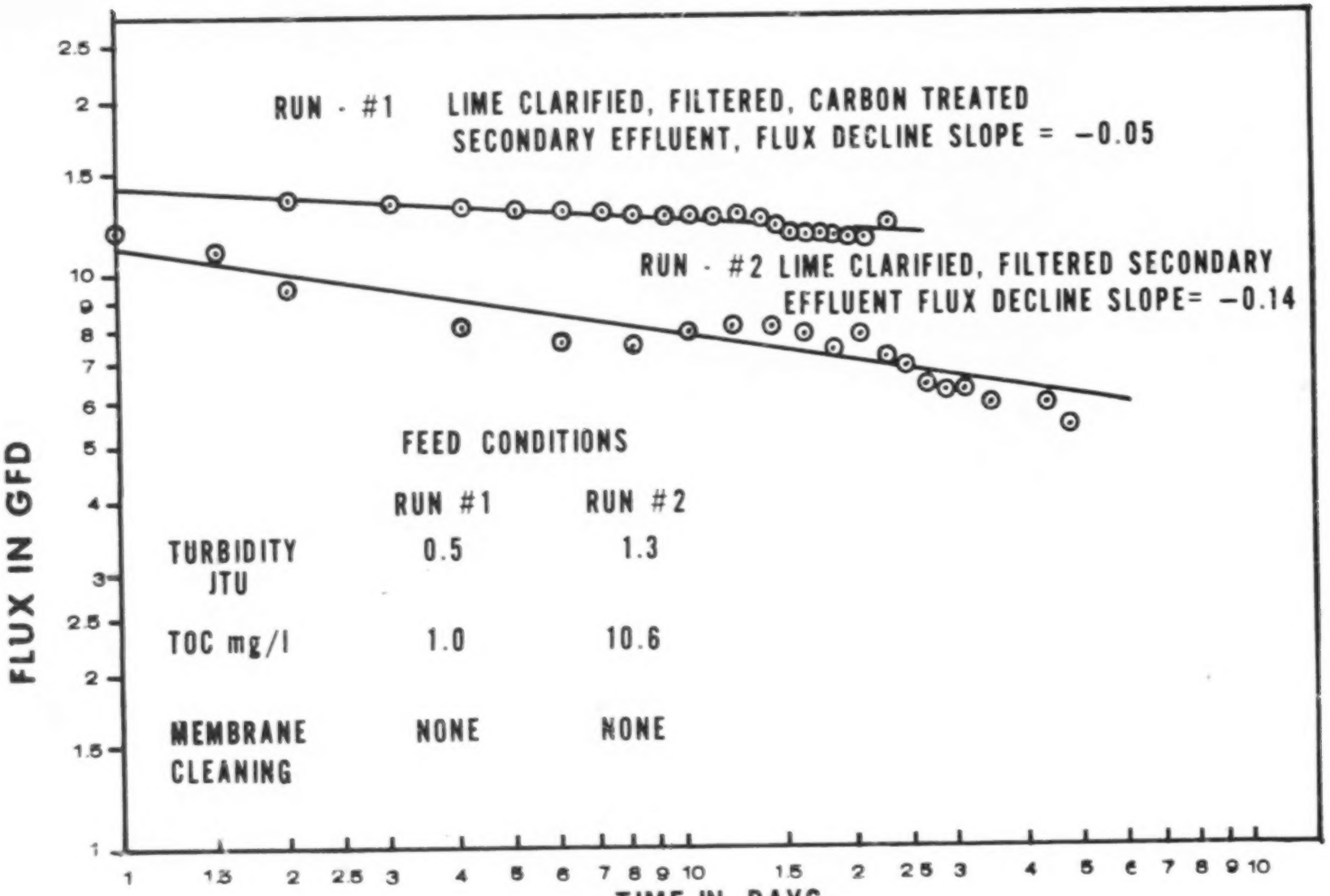

RUN - #1 LIME CLARIFIED, FILTERED, CARBON TREATED SECONDARY EFFLUENT, FLUX DECLINE SLOPE = −0.05
RUN - #2 LIME CLARIFIED, FILTERED SECONDARY EFFLUENT FLUX DECLINE SLOPE= −0.14
FEED CONDITIONS
RUN #1 RUN #2
TURBIDITY JTU 0.5 1.3
TOC mg/l 1.0 10.6
MEMBRANE CLEANING NONE NONE
FLUX IN GFD
TIME IN DAYS

FIGURE 22

COMPARISON OF FLUX DECLINE SLOPES FOR EXPERIMENTAL RUNS #1 AND #2, USING AEROJET GENERAL FLAT PLATE CELL, LEBANON, OHIO

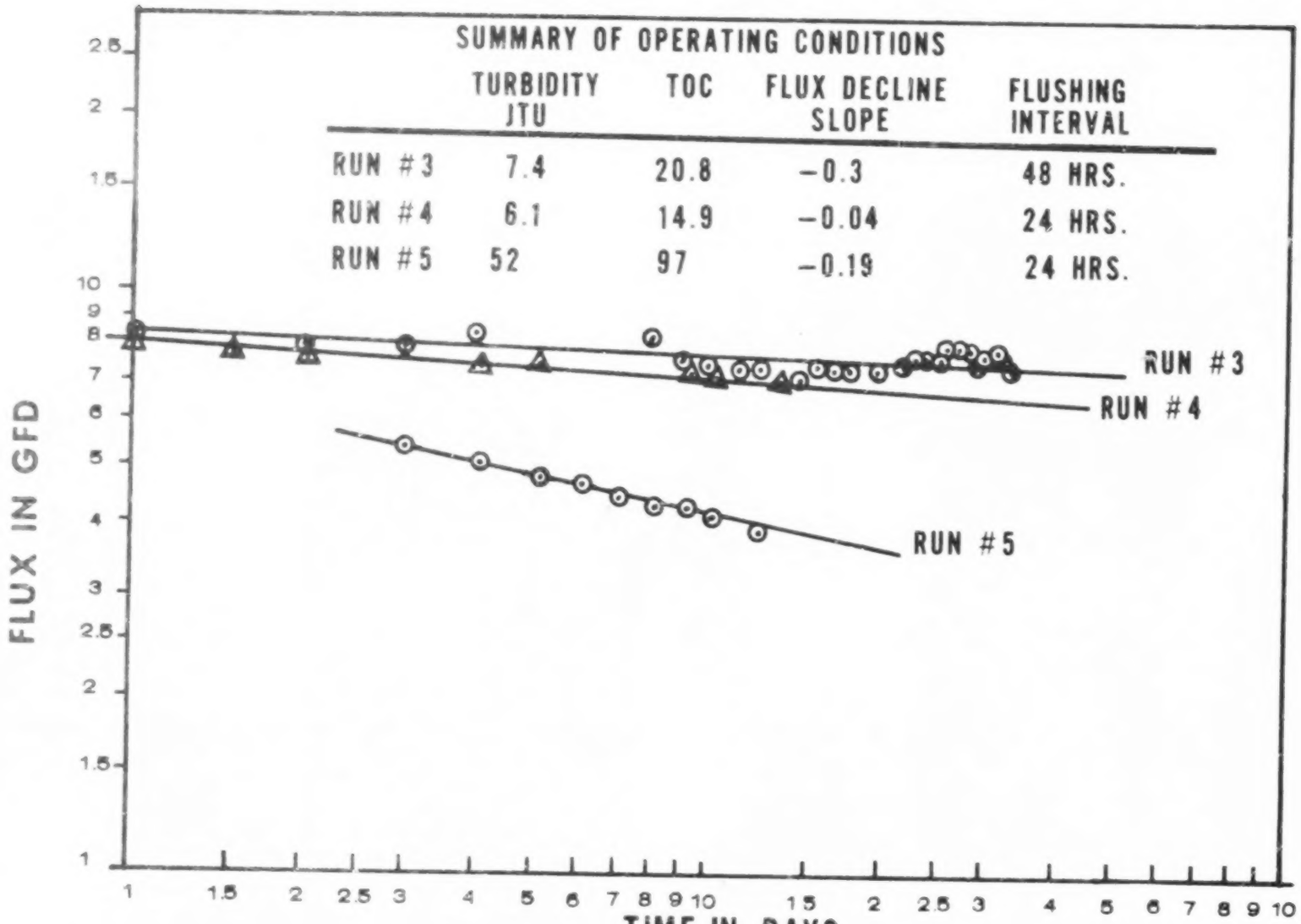


FIGURE 23

COMPARISON OF FLUX DECLINE SLOPES FOR EXPERIMENTAL RUNS 3, 4 & 5 USING AEROJET GENERAL FLAT PLATE CELL, LEBANON, OHIO

DISCUSSION AND INTERPRETATION OF PILOT PLANT RESULTS

The foregoing experiences in operating small reverse osmosis modules on variously treated waste streams have demonstrated, in all cases, that excellent separation of the contaminants of major concern can be achieved.

In general, the membranes were found capable of rejecting 93 to 95% of the total dissolved solids, 95 to 99% of the phosphate, 80 to 90% of the ammonia nitrogen, 60 to 70% of the nitrate nitrogen, 99 to 100% of the particulate matter, 90 to 95% of th total organic carbon and greater than 90% of the COD. The rejection performances exhibited by the three types of modules evaluated were similar, with the small observed differences being attributed to intrinsic differences in the membranes evaluated.

In many cases, the studies were conducted with prototype units which resulted in an initially high incidence of mechanical failures. Many of the earlier malfunctions have been eliminated by design changes resulting in much improved equipment.

All of the experiments were conducted with modules equipped with membranes designed for the conversion of "brackish water," which is characteristically low in organic content and much higher in TDS than the normal municipal wastewater streams of interest.

Work conducted early in the program showed that pH control of the feed was necessary to control the precipitation of calcium carbonate, even at moderate recovery ratios. Possible precipitation of a complex calcium phosphate at 80% recovery was noted once during the investigation, and was controlled by reducing the pH of the brine to 5.0 from an original value of 5.5. No further instance of inorganic fouling was observed during the investigation.

Organic fouling of the membranes was experienced in all of the experimental runs and can be cited as the principal cause of the flux declines that occurred. There is considerable interest and some controversy at the present time regarding the principal cause of organic fouling. The major argument centers around the correct identification of the class of material responsible for flux decline due to fouling. Most investigators agree that the material is organic in nature but dispute the particle size and size distribution responsible for the observed declines. Experimental evidence, including data reported herein, can be cited that supports equally well both sides of the issue in question. The argument is partially academic when one realizes the diverse nature of raw and treated wastewaters which are candidate feeds for treatment by reverse osmosis. These streams encompass a particle size range that varies from 10^{-4} to 10^{3} microns with molecular weights that range from less than one hundred

to several million. Attempts to characterize these waste streams as the first step in understanding the interfacial phenomena and fouling mechanism that occur at the membrane surface have not been successful, largely because of the complex nature of the waste. It is becoming increasingly clear, however, that soluble, colloidal, and suspended species are all fundamentally involved in the fouling that takes place at the membrane surface. Figure 22 shows that even a water free of gross particulate matter (turbidity of 1.3 JTU) exhibits a fouling potential sufficiently high to prohibit economic treatment by a membrane separation process unless some provision is made to control deposition of the fouling material, which can range from molecularly disperse systems through the colloidal range to the larger suspended particles.

The most successful approach in controlling the organic fouling of reverse osmosis membranes employed to date is to routinely depressurize the unit and to flush the brine sides of the membranes using the enzyme detergent flushing solution previously described. Success of the method may be due to protein hydrolysis of a slime layer that is physically deposited during operation coupled with the dispersing and cleaning action of the accompanying detergent. This concept is supported by investigations conducted at the Pomona Pilot Plant by Gulf General Atomic under FWQA Contract No. 14-12-181.

Although very little investigation of the hydrodynamic factors affecting fouling was conducted under this program, limited experiences with superficial velocity increases during the cleaning operations on the flat plate unit at Lebanon indicate some promise for this approach. Experimental investigations conducted at the Oak Ridge National Laboratory on filtered and unfiltered lake water indicate that substantially lower flux decline rates can be achieved by increasing the axial velocity in a 1/2" diameter tube. Additional studies employing turbulence promotors have shown promise of improving the efficiency of water transport through a cellulose acetate membrane (7).

A fundamental question in utilizing either increased velocity or turbulence promotors is whether these approaches will offer an economic advantage when the greater pumping cost is balanced against the increased productivity. Perhaps the greatest advantage will be realized in periodically increasing the velocity during a flushing operation. The question of membrane attrition must be dealt with and may prevent the continued use of axial velocities greater than 3-5 ft/sec (6). Preliminary investigation of fouled modules taken from the Universal Water Corporation unit operating on carbon-treated secondary effluent at Pomona, California showed some evidence of physical attrition of the membrane near the end of the module. The severity of this type of problem has not been fully assessed at this time and will require further investigation (8).

CONTRACT AND RELATED INVESTIGATIONS

In addition to the above-mentioned pilot-plant investigations, the FWQA has supported, in cooperation with other government agencies, a great deal of contract research in reverse osmosis.

This research has been directed toward three goals; the development of improved high-flux membranes tailored specifically for wastewater renovation, investigation of new methods to control flux decline due to organic fouling of the membranes, and improvements in modular design and contacting systems.

Improved cellulose acetate membranes for wastewater renovation have been developed in the laboratory by two investigators. North Star Research and Development Institute, under Contract No. 14-12-587, investigated the application of ultrathin membranes (200-2000 Å) for the renovation of municipal effluents. These ultrathin membranes were prepared on polysulfone supports and demonstrated 600 PSI fluxes of 58 to 91 GFD with salt rejections of 76 to 86% when tested in the laboratory against a 0.1% sodium chloride solution. These membranes were tested with filtered secondary effluent and demonstrated average fluxes of 17 to 38 GFD, depending on the pore size of the filter used to pretreat the feed. Evidence indicated that particles smaller than 1 micron were responsible for nearly 80% of the observed flux decline. It was also demonstrated that about one quarter of this decline could be eliminated by chemical clarification of the feed followed by passage through a 1 micron filter.

Another contractor, Aerojet-General Corporation, working with assymetric cellulose acetate films under Contract No. 14-12-553, developed three classes of membranes suitable for wastewater renovation. One membrane, a crosslinkable cellulose acetate methacrylate (CAM) membrane, synthesized by methacrylation of Eastman Type E-360-60 cellulose acetate having a degree of substitution (d.s.) of 2.09 to 2.12 to a total d.s. of 2.34 to 2.39, produced 700 PSI fluxes of 70 to 90 GFD with a TDS rejection of 80 to 90% when tested in the laboratory in flat sheet form against a synthetic feed solution.

Another membrane formulation using Eastman E-383-40 cellulose acetate as the base polymer with a d.s. of 2.28 produced 600 PSI fluxes of 64 to 105 GFD with a sodium chloride rejection of 61 to 67% when tested in flat sheet form against a 0.1% sodium chloride solution.

The third class of membranes, a blend of diacetate and triacetate, produced 600 PSI fluxes of 60 to 70 GFD with a sodium chloride rejection of 70 to 80%.

These three classes of membranes were all tested on secondary effluent and demonstrated satisfactory rejection of both organic and inorganic species of interest. The flux of these membranes was sharply reduced by organic fouling when tested without cleaning. It was demonstrated, however, that average fluxes as high as 50 GFD could be sustained when processing secondary effluent by periodically cleaning the membrane with an enzyme solution.

The above results are encouraging and indicate that, by sacrificing a small amount of salt rejection, the intrinsic flux of reverse osmosis membranes can be substantially increased. It was also shown that as the membrane flux increases the fouling problem becomes more severe.

The problem of membrane fouling has received attention from several investigators. Aerojet-General, under Contract No. 14-12-184, conducted an extensive investigation of pretreatment procedures as a means of controlling flux decline due to fouling. This study involved over ninety controlled experiments to evaluate the effects of feed quality and pretreatment on flux decline. Several classes of feed additives such as chelating agents, dispersants, and flocculating agents were evaluated on a number of waste streams including primary effluent, secondary effluent, and carbon-treated secondary effluent. In general, the flocculating agents were found to be the most successful cleaning agents, especially when followed by rapid sand filtration as a pretreatment for reverse osmosis.

Another approach taken by Gulf General Atomic, under Contract No. 14-12-181, was the examination of flushing procedures that can be periodically employed during operation to limit flux decline to acceptable levels. Gulf General Atomic first used the enzyme presoak formulations as membrane cleaning agents to aid in the removal of organic slimes. This has proven to be the most successful method presently available to maintain reasonable fluxes when treating wastewater.

Other methods of controlling membrane fouling under consideration are the periodic or sustained use of higher brine velocities, the use of integral and detached turbulence promotors, gas bubble generation at the membrane surface, generation of electric current at the membrane surface, charged membranes, and membranes containing attached enzymes. Of these methods, turbulence promotors, charged membranes, and periodic velocity increases appear most promising.

In addition to the development of high-flux membranes and fouling control techniques, there is some interest in developing more efficient modules and module configurations in an effort to lower the capital cost of reverse osmosis systems.

Developments in this area include studies of in-situ membrane replacement, cast-in-place membranes, membrane regeneration techniques, and replaceable membranes for the popular configurations. Also under study is the development of new module configurations that employ hollow fibers spun from cellulose acetate. These promise a substantial increase in module productivity, but are just entering the development stage.

RELATIVE POSITION AND FUTURE ROLE OF REVERSE OSMOSIS IN RENOVATING WASTEWATER

At the present time, the role of reverse osmosis in the reclamation of wastewater is not clear. Factors that will affect the utility of this process are three-fold; first - the need to demineralize wastewater for reuse applications; second - the economic attractiveness of reverse osmosis when compared to competing processes such as ion exchange, electrodialysis, distillation for the demineralization of wastewater on an equitable basis; and finally the potential of using reverse osmosis as a pollution control device with discharge to a receiving stream.

Since each domestic use of a water supply adds 300 to 400 mg/l of TDS it is clear that continued reuse of a single supply will require demineralization. The amount of demineralization required to maintain drinking water quality standards of 500 mg/l TDS will depend upon the TDS concentration of the source and the consumptive use ratio of the particular community. A recent study sponsored by the Office of Saline Water (9) indicates that, of the approximately 20,000 communities in the United States, about one-half are served by public water supply systems. The study further estimates that the water distributed through over 98% of these community water systems has a total dissolved solids content below the USPHS standard of 500 mg/l. Municipal water use figures in the United States indicate that the consumptive use averages about 13% (10). Wide variation in this figure occurs with as high as 50% comsumption recorded in some arid or semi-arid regions. At the latter locations, demineralization of wastewater for reuse at 90% volumetric recovery will require about 25% make-up water and a demineralization efficiency of 60% except for nitrogen.

There have been several cost estimates made for the competing processes under consideration for the demineralization of wastewater. These include ion exchange, electrodialysis, distillation and reverse osmosis. Of these, ion exchange and reverse osmosis appear to offer an economic advantage over electrodialysis and distillation. A recent cost study made by the County Sanitation Districts of Los Angeles County compared the cost, exclusive of brine disposal, of electrodialysis, ion exchange and reverse osmosis for the

demineralization of 1000 mg/l TDS water to 667 mg/l TDS (11). In this study, the feed to each demineralization process was carbon-treated secondary effluent. The total flow from the carbon adsorption columns was treated by the electrodialysis unit for a total cost of 26.9¢/1000 gallons including the cost of the carbon pretreatment. The cost for ion exchange and reverse osmosis was based on demineralizing 37% of the carbon column effluent and blending the product water with carbon column effluent to achieve a blended product quality of 667 mg/l TDS. The total cost of the blended product for these two processes including carbon pretreatment was 19.5¢/1000 gallons for ion exchange and 24.8¢ for reverse osmosis. This study indicates an advantage for ion exchange for the demineralization of carbon-treated secondary effluent. The cost for reverse osmosis used in this study was based on an average flux of 10 GFD for a membrane lifetime of two years. Using the same assumptions, but increasing the flux to 15 GFD (a demonstrated flux rate for carbon-treated secondary effluent) decreases the cost for reverse osmosis to 20.5¢/1000 gallons. In general, the cost of reverse osmosis is quite sensitive to flux rate, membrane lifetime and membrane cost. Another consideration is that, depending on the intended reuse of the demineralized water, the carbon adsorption pretreatment could be replaced with mixed-media filtration as pretreatment for reverse osmosis for an additional savings of 5¢/1000 gallons.

Consideration of the successive elimination of required pretreatments poses the question, can reverse osmosis be considered a potentially useful AWT process if demineralization for reuse is not the principal objective? It has generally been accepted that treatment of municipal wastes by reverse osmosis with discharge of product to a receiving stream is not practical. A little reflection, however, can demonstrate a potential in this area. The total costs for a conventional biological treatment system followed by tertiary processes for nutrient and residual organic removal are given below for a 10 MGD plant (12).

Treatment Step	Total Cost ¢/1000 gal.
Primary	6.2
Activated Sludge	4.8
Lime Clarification with Recalcination	7.8
Ammonia Stripping	3.6
Filtration Through Sand or Graded Media 4 gpm/sq ft	2.8
Activated Carbon Adsorption	10.2
TOTAL	35.4

Examination of the treatment sequence and associated costs suggest several possible treatment schemes incorporating reverse osmosis. Several possible combinations and the corresponding competitive costs for reverse osmosis are presented in the following table.

Pretreatment and Associated Cost		Cost for Reverse Osmosis to be Competitive ¢/1000 gal	Total Cost
Primary	6.2	29.2	35.4
Primary and Sand Filtration	9.0	26.4	35.4
Primary and Activated Sludge	11.0	24.4	35.4
Primary and Activated Sludge and Sand Filtration	13.8	21.6	35.4

This comparison clearly indicates that for reverse osmosis to become competitive, it should be employed near the primary end of a treatment train. For the situation outlined above, the application of reverse osmosis to primary or filtered primary effluent at a cost of 29.2 and 26.4 cents per thousand gallons of treated water appears competitive with present-day technology for achieving similar water quality.

The cost of brine disposal must be included in the cost of reverse osmosis treatment, and, in many cases, may prove to be a limiting factor in inland areas. A recent investigation of the disposal of brines produced in the renovation of municipal wastewater was conducted by Burns and Roe, Inc. under FWQA Contract No. 14-12-492. This study indicated that brine disposal cost varies from $0.052/1000 gallons to $0.76/1000 gallons for solar evaporation of a Colorado River water source near Denver. Brine disposal at Tucson, Arizona is estimated at $0.13/1000 gallons by deep-well injection. Another study of deep well disposal of desalination brine waste was conducted by Dow Chemical Company under OSW Contract No. 14-01-0001-1691 and reported in Research and Development Program Report No. 456. This study estimates the cost of deep-well injection to vary from 2.5¢/1000 gallons at Arkansas City, Kansas to 35¢/1000 gallons for Morgan, Colorado. The cost of inland brine disposal, whether by deep-well injection or by solar evaporation, is highly variable depending on local geologic and climatic conditions as well as on current land uses and value. In particular, disposal of reverse osmosis brines will undoubtedly require some form of pretreatment to remove the organics, especially if applied to wastewater streams such as primary or filtered primary effluents.

The reverse osmosis process, barely ten years old, developed primarily as a demineralization process is now competitive for that purpose. Present day costs of this process are prohibitively high for water renovation, unless a high premium is placed on the need to demineralize wastewater for reuse. Recent emphasis by FWQA in supporting development of membranes and membrane systems specifically designed for wastewater treatment, the progress being made in controlling organic fouling, and technological improvements being made by the industry itself should substantially improve the posture of reverse osmosis in the foreseeable future. Widespread use of this process will ultimately depend, of course, on the economic advantage that will be demonstrated over competing processes in any particular treatment scheme.

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Municipal Treatment Research Program
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6 Title: RENOVATION OF MUNICIPAL WASTEWATER BY REVERSE OSMOSIS

10 Author(s):
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*Reverse osmosis, *Desalination processes, *Membranes, *Demineralization, *Semipermeable membranes, osmosis, separation techniques, pressure, membrane processes, water reuse, water renovation, wastewater treatment, fouling, organic fouling

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*Ultrafiltration, *Cellulose acetate membranes, *reverse osmosis modules, Hyperfiltration, Water conversion, Reverse osmosis modules tubular, Reverse osmosis modules spiral wound, Flux, Flux decline

27 Abstract:
The increasing demand for clean water makes clear the need for improved treatment of municipal wastewater. Deliberate reuse of wastewater requires efficient removal of "residual" organic and inorganic solutes. A common and effective approach has been to supplement biological processes with chemical and physical processes that, together, are capable of achieving water quality consistent with reuse requirements. A more direct approach is to use a single process such as reverse osmosis which exhibits a potential to separate a broad spectrum of contaminants from primary and secondary effluents.

The pilot plant studies reported herein were addressed toward defining the problems associated with the reverse osmosis processing of variously treated wastewaters. The effects of feed water quality on membrane performance were evaluated, as were methods to control membrane fouling. A comparison of three configurations; flat plate, spiral-wound, and tubular was made to determine their relative merit in treating wastewater. Finally, a cursory examination of costs was made.

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